VOL. 80 No. 2048
OCTOBER 11 1958

CHEMICAL AGE

Telephone: FLEet Street 3212 (26 lines)
Telegrams: Allangas - Fleet - London

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IN THIS ISSUE

Interfacial Polycondensation	590
Development in 'Living' Polymers	591
Laporte's New Peroxide Plant	591
ISR's Three Grades of Rubber	591
Distillates	592
New Survey on Chemical Engineers	593
Polarography Symposium	595
New Process for Calcium Metaphosphate	599
Pfizer's Acquire Kemball, Bishop	600
Overseas News	601
Analytical Review	603
Cyclopentanone Now Available	604
Diary Dates	604
Market Reports	604
Commercial News	605
Trade Notes	606
People in the News	607
New Patents	608

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US RECOVERY

WHEN the US recession was at its height early this year, and prognostications were of the gloomiest, several leaders in the US chemical industry were emphatic in their opinion that by the fourth quarter, a definite upswing would be noted all round (see CHEMICAL AGE, 26 April, 1958, page 763). That this opinion was correct is now indicated from present news of the US chemical industry.

Conditions are reported to be improving almost everywhere. The last quarter, the US chemical industry believes, will be an excellent one, due to unexpectedly good sales during the summer months. Demands are reported to be rising and in some instances exceed present stocks. Two examples quoted at the National Industrial Conference in New York recently by W. Ward Jackson, vice-president sales, of Commercial Solvents Corporation, were ethanol and isopropanol. Depleted inventories and a return to 'normal' buying habits are now apparent.

On Wall Street, chemical stocks are noted as starting to climb, although the response varies somewhat. Drugs and pharmaceuticals are still in the lead, and are stated to be up by about 60 per cent from October 1957 figures. Moderate size chemical producers have stayed with general market trends, and at the end of last month reached a new record level since their 1956 peak period. It is the stocks of some of the larger chemical companies that are showing a slower rise, although shares of one or two such companies have shown gains during recent weeks.

This encouraging improvement in US chemical companies' shares is due to a return to increasing production and sales, while excess inventories are no longer the general rule. Cost cutting programmes have also had a noticeable effect. The feeling in Wall Street is that compared with other industrials, US chemical shares are not overpriced. Particularly promising stocks are those of industrial gases, plastics, paints, and basic chemicals required by manufacturers of steel or consumer goods. Problems now associated with chemical companies are reduction of costs, over-capacity, competition and over-diversification.

It is expected that improvements in the industry will be ensured, for more general construction, and housing plans for 1959 should mean a greater need for plastics, for structural and protective purposes; a 22 per cent improvement over 1958 in the car industry influencing chemicals such as additives, ethylene glycol, synthetic fibres and rubbers; a 75 to 80 per cent capacity fulfilment in the steel industry instead of the 47 per cent earlier this year; an increase in defence spending with consequent calls on rocket fuels, plastics etc.; restoration of consumer confidence, leading to increased calls for synthetic fibres and plastics.

Chemicals quoted as being in short supply are helium, naphthenic acid, dipentaerythritol, crude glycerol and chlorine. There is, however, excess capacity in certain sectors of the chemical industry, notably in fertilisers, plasticisers and penicillin.

'Selective' expansion programmes appear to be continuing at a steady pace. Some companies have had to spend large sums on plant and equipment

as a result of new product developments, although existing plants are running below capacity. There has also been, and continues to be, heavy expenditure by US chemical manufacturers on modernisation of plants and factories in order to improve profit margins. The recession has indeed brought home the value of concentrating on the more profitable products, of improving sales techniques etc.

While expansion programmes have been carefully reviewed by US chemical manufacturers, in some instances leading to cut-backs and a slowing down in completion of projects, a number of companies have announced new construction projects. One of these, Air Reduction Chemical, are now building a 20 million lb. a year polyvinyl resin plant at Calvert City, Kentucky, and a supporting pilot plant in New Jersey. Union Carbide are to set up two units at their Seadrift, Texas, plant with a capacity of 100 million lb. of esters and alcohols. Diamond Alkali recently announced plans for a multi-million dollar research installation, and Stauffer Chemical have completed plans to construct the first plant to make Eptam herbicide.

Plans for new ammonia and urea plants have been announced by Cooperative Farm Chemicals Association, Lawrence, Kansas, which will raise CFCA's ammonia capacity by 100 tons a day and urea capacity by 30 tons a day. Valley Nitrogen Producers are also going ahead with their 150 ton-a-day ammonia plant near Fresno, which is scheduled for completion in November of next year.

An air of cautious optimism prevails in the US at present, but it is believed that there will be some bad patches in 1959 where over-production and bad selling practices exist. At the same time, the world political situation is kept very much in mind, as it could change the present US recovery pattern. In the event of any crisis, chemicals are likely to be 'frozen'. Those chemicals with domestic outlets would obviously become scarce.

INTERFACIAL POLYCONDENSATION

SEVERAL US research groups, notably E. I. Du Pont de Nemours, Celanese Corporation of America and General Electric, are exploring the possibilities of interfacial polycondensation, and at the recent symposium on Polyethers and Condensation Polymers of the Division of Polymer Chemistry at the 134 ACS National Meeting. (*Chem. and Engng. News* 1958, **36,** No. 37, 52.) Du Pont's Pioneering Research Laboratory have indicated their findings to date. Compared with melt polycondensation as now mainly used for condensation polymers interfacial polycondensation is stated to use simpler equipment and requires conditions described as more nearly atmospheric. Potential attractiveness of the process for large-scale polymerisations is that polymerisation using monomers, e.g., diamines or diacid chlorides is irreversible. Interfacial polycondensation is stated to work particularly well with polyamides, polyurethanes and polyphenyl esters.

According to Du Pont workers, E. L. Wittbecker and P. W. Morgan, high monomer purity, exact balance of reactant monomers and quantitative yield are not required to make high polymers. Interfacial polycondensations are reported as being less hindered by side reactions in obtaining high molecular weights—a problem associated with melt polycondensation. Yields of high molecular weight polymers using interfacial polycondensation have ranged from 5 to 100 per cent. Polymers are produced in several minutes' reaction time, at room temperature or below, and at atmospheric pressure. Also no special equipment is necessary.

Polyamides are reported as being the most easily prepared by Du Pont researcher R. G. Beaman. High melting and difficultly soluble polymers are stated to be harder to make in very high molecular weights. Cyclic secondary amines, such as piperazine and its derivatives have been found to give high molecular weight polyphthalamides by M. Katz. These polymers, when cast from solution in organic solvents (e.g. chloroform), produce clear flexible films. Failure to obtain high molecular weight polyphthalamides is considered by Katz to be due probably to end-grouping by imide formation.

Polysulphonamides are made with disulphonyl chlorides and both aromatic and aliphatic diamines, reported S. A. Sundet, W. A. Murphey and S. B. Speck. Only low molecular weight polymers have been produced from aromatic diamines, due, it is believed, to the low basicity of aromatic diamines together with lower reactivity of sulphonyl chlorides compared with carboxylic acid chlorides.

A wide variety of polyurethane structures has been produced by combining bio-chloroformates and almost every type of diamine used in condensation polymers. High molecular weight polymers have been prepared from ethylenediamine and high molecular weight polyurethanes from aromatic diamines and cyclic secondary diamines.

Interfacial polycondensation has been used by J. R. Schaefgen, F. H. Koontz and R. F. Tietz to make polymers from monomers having both functional groups in a single molecule. Polyamides and polyurethanes have been produced from amino acid chlorides and aminoalkyl chloroformates respectively, by blocking the amino groups by salt formation until the reaction is ready to begin. The salt is formed by use of hydrochloric acid or *p*-toluenesulphonic acid.

Problems are associated with monomers as salts. They are difficult to purify as they must be recrystallised from a non-reactive dry solvent, and some monomers are insoluble in such dry solvents as benzenes, chloroform or dichloromethane. Insoluble monomers salts can react when dispersed as a suspension in the organic phase or in water they dissolve and hydrolyse. This can be avoided by using high concentrations of salts such as potassium carbonate to 'salt' the monomer into the organic phase.

Another worker, W. M. Eareckson III, reports that interfacial polycondensation is very suitable for producing high molecular weight polyphenyl esters from diphenols and dicarboxylic acid chloride. He has found that, in general copolymers of two bisphenols and one diacid chloride are prepared easily; negatively substituted bisphenols tend to give lower molecular weight polymers; aliphatic diacid chlorides work only moderately well in giving high molecular weights; and, aliphatic glycols are too unreactive under interfacial polycondensation conditions to give polyesters.

Variables of interfacial polycondensation affect the final polymer and also the reaction, stated Morgan and Stephanie L. Kwolek. In the laboratory vigorous stirring is used, but polycondensation can be effected without stirring as when an aqueous solution of hexamethylenediamine is placed upon a carbon tetrachloride solution of sebacoyl chloride. A film of high polymer forms at once at the interface of the liquids which can be pulled out continuously.

Reaction rates of unhindered aliphatic acid chlorides with primary diamines are estimated at about 10^4 litre-mole^{-1} sec.$^{-1}$. The limiting rate step is stated to be the transfer of diamine across the interfacial boundary.

It is widely held that much more will be heard about interfacial polycondensation as a simple, rapid, low temperature route to a wide variety of polymers.

Novel Polymerisation System Produces 'Living' Chains

New Work Described at SCI Meeting

AN interesting new development in polymer chemistry—a novel type of polymerisation system in which active or 'living' chains are produced—was described by Professor M. Szwarc of Syracuse University, US, at a recent meeting of the Plastics and Polymer Group, Society of Chemical Industry.

There are three stages in the formation of a polymer molecule: initiation, propagation and termination. In free radical polymerisation and likewise in the case of carbonium ion-activated ionic polymerisation, it is impossible to prevent the termination stage occurring. The polymerisation initiated by negative aromatic ions, like naphthalene", and carried out in non-proton donating solvents, like tetrahydrofuran, leads to 'living' polymers, which are capable of growing if a monomer is available. Such polymers can be eventually 'killed' by adding, e.g., a drop of water, which transfers a proton to the active carbanion ends and turns them into inactive groups.

Initiation by Negative Hydrocarbon Ions

The initiation by negative hydrocarbon ions results from an electron transfer to the monomer (I and II) and eventually the dimerisation of the primarily formed species produces polymers with both ends living (III).

$$:CHX\text{-}CH_2\cdot \text{ (I)} \qquad \cdot CHX\text{-}CH_2: \text{ (II)}$$
$$:CHX\text{-}CH_2\text{-}CH_2\text{-}CHX: \text{ (III)}$$

Professor Szwarc illustrated this theme with reference to the polymerisation of styrene in tetrahydrofuran using sodium-naphthalene complex to initiate polymerisation. Polymerisation, if carried out correctly, always goes to completion. The green colour of naphthalene-ion instantaneously changes into deep red on addition of styrene, the latter colour due to styrene-ends. After completion of polymerisation the red colour persists, indicating that there is no reverse reaction. If an additional amount of styrene is added after completion of the polymerisation of the first portion, the polymerisation starts again and the reaction again goes to completion. Viscosity measurements have proved conclusively the existence of living ends in these polymers.

Among various intriguing possibilities, the technique gives rise to a novel method for the preparation of block polymers. After completion of the first polymerisation stage a second monomer is added to the still living polymers and the addition of alternate monomers continued as each stage is concluded. By varying the quantities of monomers employed in each step and the monomer:catalyst ratio in the initial step, block co-polymers may be 'tailor-made'. The living polymers are killed by adding a drop of water, the introduction of carbon dioxide or other proton donors with the formation of a chemically active group on the end of the hydrocarbon chain.

Extreme care must be taken in performing these experiments since minute amounts of impurities left in the monomer, or the solvent, or absorbed on the walls of the reaction vessel, can ruin the investigation. It has been found in the case of heterogeneous catalysis of ethylene, however, where similar contamination problems are encountered, that the high order of purity demanded can be more readily obtained in larger scale operation. It will be interesting to see how quickly the experimental difficulties can be overcome and this fascinating technique developed into a commercial process. Will it be used to make polymers of controlled molecular weight, block co-polymers of tailor-made character, or low molecular weight polymers (of narrow molecular weight distribution) with reactive end groups?

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Three Synthetic Rubbers from New Fawley Plant

FIRST three grades of general purpose synthetic rubbers to be made by the International Synthetic Rubber Co. Ltd. at their new Fawley plant will be marketed under the trade name Intol.

The first, Intol 1500, with excellent physical properties, is a staining rubber suitable for tyres hard rubber, etc. The second, Intol 1502, is of similar quality to Intol 1500, but is designed to be almost colourless and therefore suitable for white side-walls of tyres, sports goods, floor coverings, etc., as it does not transfer any colouring to the finished product. The third, Intol 1710, has been extended at the latex stage by the addition of an aromatic oil. With easy processing characteristics it is suitable for a variety of moulded and extruded goods.

The plant, with a rated capacity of 50,000 tons a year, copolymerises butadiene and styrene at about 40° F. The two raw materials are supplied in liquid form and polymerisation takes place in an emulsion. An improved redox catalyst system makes it possible to polymerise about 2-3 hours quicker than is the case with 'hot' rubbers.

After polymerisation, the latex is passed through a recovery unit where butadiene is removed; styrene is removed by vacuum steam distillation.

Fuller details of this new plant will appear in CHEMICAL AGE next week.

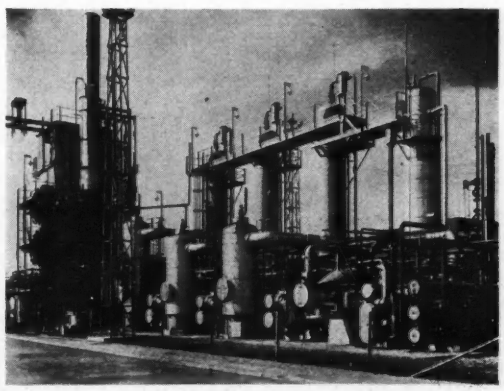
Laporte's New-Process Peroxide Plant at Warrington is World's Largest

A NEW hydrogen peroxide plant believed to be the largest in the world—and certainly the first of its type to be erected in Europe—has been set up at the Warrington, Lancs, works of Laporte Chemicals Ltd., and is now on stream.

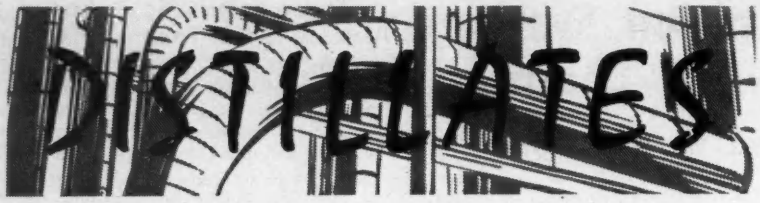
Production is by a new 'autoxidation' process developed in Laporte's own research department, hydrogen being extracted from butane by thermal extraction in a Power-Gas/Hercules high-purity plant and reacted with water.

The butane is obtained from a neighbouring petroleum refinery. The Power-Gas/Hercules installation consists of a reforming furnace followed by three stages of CO conversion and CO_2 removal with final methanation. The additional stage of conversion is to reduce the final residual methane to a minimum.

The hydrogen is stored in a Wiggins 'dry seal' gasholder from which supplies are drawn for the 'autoxidation' plant.



The high-purity hydrogen unit installed at Warrington by Power-Gas to supply Laporte's new 'autoxidation' hydrogen peroxide plant



★ THE OIL and oil engineering industry have no need to fear that they will be displaced in a few years by the atomic energy programme. Competition will be negligible over the next 20 or 25 years, although there may be a reorientation of the relative amounts and nature of products derived from petroleum distillation.

That was the theme of the after dinner speech made by Dr. James Taylor, director of ICI and chairman of Yorkshire Imperial Metals, when he proposed the toast of the Council of British Manufacturers of Petroleum Equipment at the annual dinner last week. In fact he felt that oil was too precious a substance to be burnt and should be kept for the production of petrochemicals.

He referred to a US estimate that output of petroleum-derived chemicals had risen from 250,000 tons in 1940 to more than 6 million tons in the last few years. Total European investment in plants now stood in the region of £125 million and in the UK alone was scheduled at the end of 1958 at about £100 million.

Dr. Taylor, who joined ICI Nobel Division research department 30 years ago, said that although chemical technology was advancing rapidly so also was the technology of materials of construction. The new metals should provide the means of fulfilling equipment needs which would have been impossible a short time ago.

★ EVIDENCE of the increasing, if belated, respect in which industry holds research comes from the Aston Chain and Hook Co., non-ferrous metal engineers. The opening of their new £15,000 laboratory block at their Erdington (Birmingham) works, is stated to give the 'lab' a more than usually prominent role in the organisation's activities.

So far from being a narrow and specialist department, as is often the fate of the industrial laboratory, the new block has now become the headquarters of a development department completely integrated with every form of works and staff activity from process control to work study.

Expressing an enlightened view, Mr. Noel Bond-Williams, managing director of the company, tells me 'This, even more than my own office, is the nerve centre of the factory, ready to tackle every kind of problem. The old idea of a laboratory as a sort of ivory tower inhabited by men in white coats peering into microscopes and with little practical knowledge of management or shop floor conditions is hopelessly out of date.'

Manager of the new development department, which is responsible for process control, inspection, work study, factory layout and product and process development work, is 28 years' old Mr. Norman Harvey, formerly the company's chief metallurgist. Mr. Harvey qualified for his M.Sc. after being seconded for a year to take a full-time course in engineering production at Birmingham University.

★ FOLLOWING the CHEMICAL AGE report two weeks ago that nitrogen is being considered as an aerosol propellant in this country, I was interested to see in *Packaging Abstracts*, published by Patra, a reference to survey of aerosol food-packing in the US. It appears that aerosol containers using the nitrogen propellant system have been found to suffer almost undetectable weight loss over an 18 month period.

New products being tested for aerosol packaging are liquid coffee and chocolate syrup. The same journal refers to what is claimed to be the first aerosol inhalant preparation and to a rechargeable aerosol container which achieves complete separation of propellant and product during storage; since only gas is drawn off from the liquefied propellant during operation, dispersal of the propellant is eliminated. The aerosol 'shell' can then be refilled with product and a propellant cartridge.

German work noted includes a new propellant pack for starch, the use of vinyl chlorides and mixtures of propane and butane. News is given of the use of an aerosol for a highly viscous motor polish, the high wax content of which is maintained in suspension form and in chemical equilibrium with the propellants. A Chicago firm claims to have adopted gas chromatography for the analysis of volatile components in pressurised products.

★ HOW NYLON filter cloths helped Richard Thomas and Baldwins prevent pollution at their Ebbw Vale works and conserve water which is used to clean gases from the blast furnaces, is the subject of a note sent to me by British Nylon Spinners. These gases are cleaned by three stages: a dustcatcher, water towers and electro-static precipitators.

The used water (4,000 g.p.m.) from the towers is fed to Dorr Thickeners where solids sink to the bottom and are propelled to the centre of the 'settling ponds' where they are pumped away in the form of sludge. Originally this sludge was discharged into a nearby river. The new pollution regulations will make this illegal. The problem was intensified because of extra demands for water which had to come from a more efficient use of existing quantities.

Filtration was the obious answer and a plant of three disc filters was installed. The first of its kind in the UK, it presented many difficulties before it operated efficiently. Various filtering mediums were tried before nylon was found to be the answer. By using four discs on a single filter more than two tons of cake with a moisture content of about 30 per cent are moved in an hour. Although the material is highly abrasive, consisting of more than 50 per cent spent iron oxide, the nylon cloths have a life of between three to four months. The fabric used is a 12-oz./sq. yd. continuous filament twill.

★ RECENT proposals to establish a petrochemical industry based primarily on synthetic rubber production (see CHEMICAL AGE, 13 September, page 438) were commented on by Sir Daniel McVey, chairman of Dunlop Rubber (Australia) at the company's annual meeting in Melbourne. Sir Daniel said that three leading rubber companies in Australia—Dunlop, Goodyear and Olympic—had concluded that locally produced synthetic rubber would sell for substantially more than the current landed cost, including duty, from the UK, the US, or Canada.

When the annual quantity of synthetic rubber needed by the Australian tyre industry becomes sufficient the three companies will immediately consider plans for joint manufacture in Australia.

I said earlier that competition to establish chemical units was growing. Sir Daniel, I see, indicates that it is difficult to understand the attitude of governments which encourage and endorse applications for overseas capital as in the proposed establishment by B. F. Goodrich Co., US, of a £A3 million tyre factory. Australia, he says, has to guard against the wrong application of available funds, the over provision of manufacturing capacity in well-established and economically operated industries and unnecessary duplication of capital.

★ TWICE this year the protective value of Sorbsil has been rigorously tested in both the most humid and the coldest of climates. First with Fuchs and Hillary in the snow and ice of Antarctica and later in the heat of South American jungles.

The Cambridge Northern Amazon Expedition, which returned to this country last month after 10 weeks in south British Guiana and northern Brazil, found that an 8 oz. bag of Sorbsil, in a thick polythene bag closed by a rubber band and placed in a tin, kept cameras and binoculars dry for 6-8 weeks without recharging.

When the expedition had finished a roll of film, it was dried out over the gel before sealing up and sending to the UK for processing. Needles, razor blades, food and clothing were all preserved, providing the clothes were dry before placing in the bags with the gel. Those with cameras not protected in this way found that lenses were covered in fungi and the films scarred by damp.

Alembic

Next Meldola Award Due Early In 1959

NEXT AWARD of the Meldola medal by the Royal Institute of Chemistry will be made early in 1959 to the chemist who, being a British subject and under 30 years of age at 31 December 1958, shows the most promise as indicated by his or her published chemical work brought to the notice of the council before the end of 1958.

No restrictions are placed on the kind of chemical work or the place in which it is conducted. The merits of the work may be brought to the notice of the council either by persons who desire to recommend the candidate or by the candidate himself, by letter addressed to the president, RIC, 30 Russell Square, London WC1, the envelope being marked 'Meldola Medal'.

The letter should be accompanied by six copies of a short statement on the candidate's career (date of birth, education and experience, degrees and other qualifications, special awards, etc., with dates) and of a list of titles, with references, of papers or other works published by the candidate, independently or jointly. Candidates are also advised to forward one reprint of each published paper of which copies are available.

Humglas To Build Gas Plant For Carbide Kilns

Two 7-ft. diameter mechanical gas producers are to be built by Humphreys and Glasgow Ltd., Humglas House, Carlisle Place, London SW1, to supply gas for firing lime-kilns at Carbide Industries' new works near Londonderry, where calcium carbide and acetylene are to be produced. The acetylene will be supplied to du Pont (UK) Ltd., for making neoprene.

Gas-firing of the kilns has been chosen to provide an efficient use for by-product gases from other parts of the works supplemented with gas from the new producers.

The Carbide Industries' works are at Maydown, on Lough Foyle, a few miles from Londonderry, near a new EBNI power station and the du Pont factory. The CIL site covers a total area of some 50 acres and the factory, when in operation, will employ about 300 workers.

Two New Bradford Courses

Two special short courses have been arranged by the Department of Chemical Technology, Bradford Institute of Technology. On Mondays, from 6 October to 8 December, at 7.30 p.m., a course will be held in 'Manufacture and dyeing of man-made fibres and plastics.' On 31 October and 1 November there will be a course in 'Techniques of Polymer Chemistry'. Fee for each of these courses will be £2 5s 0d.

International Patents and Trade Marks Convention in Lisbon

Revision of the International Industrial Property Convention on Patents and Trade Marks is being considered by a diplomatic conference which began in Lisbon on 6 October. Fifty member countries are attending. Leader of the UK delegation is Sir Stephen Holmes.

Fewer Chemical Engineers On Plant Work, More In Admin.

PROPORTION of chemical engineers employed in administration in the UK last year was 30 per cent of the total, against 20 per cent in 1953. The proportion engaged in plant operation was almost halved over the same period, declining from 23 per cent in 1953 to 12 per cent last year.

The proportion employed on research and development, 22.7 per cent, showed little change compared with 1953, but compares with a US proportion of 31 per cent. The percentage of chemical engineers employed on design, construction, installation, 21 per cent in 1957, is much higher than the latest known figure for the US, 7 per cent.

These figures are given in a survey of the supply and distribution of chemical engineers in Britain, carried out by the Institution of Chemical Engineers. Based on returns made by members towards the end of 1957, the survey shows that the greatest concentration of chemical engineers is in the mineral oil refining industry, followed by the chemical and allied trades.

The survey states that the mineral oil industry has been more successsul than some in building up an adequate corps of chemical engineers, while some industries do not appear to have a proper appreciation of their needs for chemical engineers.

It is pointed out that the Report on Scientific and Engineering Manpower in Great Britain gave the total number of qualified chemical engineers in the UK in 1956 as 1,500 and the total number needed by 1959 as 2,200. An estimate made on behalf of the Institution showed, however, that the total population of qualified chemical engineers in the UK in 1956 should have been of the order of 5,000 and that this should increase to 10,000-11,000 by 1966.

Output of qualified chemical engineers from UK universities and technical colleges totalled 277 in 1956 and is estimated to be 940 by 1966. The 1966 output of 900 would meet the needs of expansion and replacement, but it was unlikely that the shortage of qualified men would be made good by that time.

The 1956 apparent shortage—the difference between 1,500 and 5,000—did not mean that there are or were 3,500 vacant chemical engineering posts, for a number of these positions are held by those with some chemical engineering experience, but who are not qualified. The need for more intensive and detailed planning before construction is started made heavy demands on qualified men and there was obviously no room for complacency.

Concentration of Chemical Engineers

Industry	Estimated Total No. of chemical engineers	No. of qualified scientists and engineers	No. of adult male workers
Mineral oil refining inc. petro-chemicals ...	236	1,303	32,210
Chemicals and allied trades (Coke ovens and by-products work, chemicals and dyes, fine chemicals inc. pharmaceuticals, explosives, paint, varnish, soap, synthetic detergents, other oils)	806	8,234	342,430
Textiles	56	846	50,790
Gas	60	1,775	121,950
Metal manufacturers(non-ferrous)	21	1,128	96,550
Food, drink and tobacco	72	1,664	471,970

Analysis of I. Chem. E. Membership by Industry

	Returns	1957 (%)	1953 (%)
Coke ovens and by-products works	37	2.1	2.8
Dyes & dyestuffs	36	2.1	2.6
Fertilisers	26	1.5	1.8
Synthetic resins & plastics ...	76	4.4	4.9
Heavy chemicals: Inorganic ...	124	7.2	12.2
Organic	104	6.0	3.6
Industrial gases	26	1.5	0.6
Fine chemicals inc. pharmaceuticals	104	6.0	5.0
Explosives	40	2.3	2.9
Paint, varnish	15	0.9	0.9
Soap, synthetic detergents, glycerine, etc.	43	2.5	2.6
Mineral oil refining inc. petro-chemicals	189	11.0	11.2
Other oils, greases, glues ...	15	0.9	2.6
Chemical plant manufacture	257	14.9	14.4
Instruments	8	0.5	0.5
Treatment of non-metalliferous mining products, other than coal	18	1.0	1.3
Metal manufacture: Ferrous	16	0.9	0.7
Non-ferrous	17	1.0	2.4
Textiles (inc. man-made fibres)	45	2.6	3.5
Food, drink, tobacco ...	58	3.4	2.5
Paper, paper board	21	1.2	0.6
Rubber	6	0.4	0.5
Atomic energy	90	5.2	—
Gas	48	2.8	2.6
Electricity	9	0.5	0.7
Coal	21	1.2	1.8
Water	—	—	0.2
Sewage treatment	5	0.3	0.5
D.S.I.R. research stations, research associations, etc. ...	19	1.1	0.9
Government service ...	24	1.4	0.6
Private practice	67	3.9	2.8
Teaching	94	5.5	3.0
Others	66	3.8	6.8
	1,724	100.0	100.0
Retired and no returns ...	452		
	2,176 (79.2% returns)		

Analysis of I. Chem. E. Membership by Occupation

	Members	Associate Members	Graduates	Total	1957 (%)	1953 (%)
Administration and management	176	264	69	509	30.0	20.2
Plant operation, production and plant maintenance	18	109	81	208	12.3	22.8
Design, construction, installation, commissioning	40	200	115	355	20.9	20.4
Development and research	57	190	138	385	22.7	21.8
Technical sales and service	14	32	14	60	3.5	2.0
Teaching	24	36	27	87	5.1	3.0
Consulting	56	18	—	74	4.4	3.0
Others	2	6	10	18	1.1	6.8
	387	855	454	1,696	100.0	100.0
No return or retired members	112	201	167	480		
	499	1,056	621	2,176		

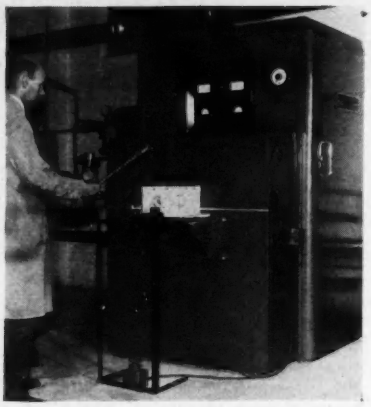
New Hot-forging Plant for Stainless Steel Bolts

THE 81-year-old firm of Sandiacre Screw Co. has recently installed a hot-forging plant at their Sandiacre, Derbyshire, factory. This decision was taken after long and careful research into the best of the European hot-forging techniques.

For the high frequency induction heating of stainless steel, Sandiacre Screw have installed a furnace by Wild-Barfield. This machine provides localised and faster heating than an open furnace. It also prevents distortion and reduces scaling. But the basic factor which prompted the company to install the Wild-Barfield was the automatic control of correct forging temperature. Another outstanding feature is that the machine will heat a $\frac{5}{8}$ in. billet to 1,150°C in eight seconds.

Hardening, tempering, brazing and soldering operations of a mass-production character can also be done on this machine, and equipment can be changed over quickly from one type of operation to another. It can if required be used for general heat treatment work in departments dealing with a wide variety of jobs. The Wild-Barfield furnace leaves nothing to the human element as in open furnaces.

The fact that the correct forging temperature is controlled automatically means that production is uniform and the desired convex grain flow always exists in the bolt head and the clean heat provided by the furnace prevents undue oxydisation and scale formation is kept to a minimum.



Wild-Barfield induction furnace which heats a $\frac{5}{8}$ in. stainless steel billet to 1,150°C in eight seconds

The new installation will reduce the prices of stainless steel bolts to UK consumers. Also it is estimated that it will save the firm 70 tons of stainless steel per year, thus avoiding the wastage of a scarce metal containing elements purchased from dollar markets.

Hilger Direct-readers for the Analysis of Iron and Steel

RECENT research has shown that Hilger medium direct-readers can be used successfully to determine (a) silicon, manganese and the principal residue elements in mild steel, (b) silicon and manganese in cast iron, and (c) certain elements in low-alloy steels if the concentration of the main alloying constituents is not too high.

Results of analysing a single representative sample of mild steel (steel with up to 0.25 per cent carbon) are quoted by Hilger and Watts Ltd., in a research report—BR7. In the following table the standard deviations were obtained from ten successive sparkings:

Element	*Content % & Standard Deviation (s. d.)*	*Coefficient of Variation on Content %*
Silicon	0.107 ±0.0024	2.2
Manganese	0.49 ±0.11	2.2
Chromium	0.165 ±0.0046	2.8
Cobalt	0.06 ±0.0040	6.6
Copper	0.21 ±0.0084	4.0
Iron	(Internal standard)	
Molybdenum	0.095 ±0.0020	2.1
Nickel	0.080 ±0.0063	7.9
Tin	0.04 ±0.0021	5.2
Titanium	0.035 ±0.0024	6.9
Vanadium	0.05 ±0.0015	3.0

The following results were obtained from the analysis of cast iron:

Silicon	1.54 ±0.028 (s. d.)	1.8
	1.54 ±0.029	1.9
Manganese	0.76 ±0.012	1.9

In the case of low-alloy steels, the results are reported as being very much better than might have been expected from the comparatively low dispersion of the instrument. The best all-round discharge was given by 40-μF, 0.5 mH, zero resistance setting on the Hilger BNF source.

Smoke Density Telescope Shown at Llandudno

A NEW portable instrument which should be of value to local government smoke inspectors is a smoke density telescope designed at the Fuel Research Station of the Department of Scientific and Industrial Research. Tests have shown it to be a simple means of assessing the density of plumes of smoke issuing from chimneys—and more convenient to use than the standard Ringelmann charts.

It was shown at the exhibition in Llandudno, held in conjunction with the annual conference of the National Society for Clean Air, from 1-4 October.

Smoke can be viewed through the telescope and its density compared with two translucent screens fitted in the body of the telescope and corresponding to No. 2 and No. 3 Ringelmann shades.

Enjay Butyl for Use in Appliance Applications

RESISTANCE of Enjay butyl rubber to chemicals such as detergents rinse aids, ozone, etc., is noted in Bulletin No. 7 issued by Esso Petroleum Co. Ltd., Chemicals Department, 36 Queen Anne's Gate, London SW1. Typical Enjay butyl formulations designed for extruded and moulded applications, such as washing machine hose, boots, gaskets, etc. are described.

Production of drain hose requires a stock that will process well, give a smooth tube, have high hardness, good ozone resistance and possess excellent resistance to tear. Such a hose stock is stated to be as follows:

Enjay Butyl 150	100 parts	Zinc oxide	5
SRF black	75 ,,	Stearic acid ...	1
FEF black	45 ,,	Sulphur	1.5
Petrolatum	5 ,,	Mercaptobenzothiazole	2
Paraffin wax	5 ,,	Tetramethylthiuram disulphide	1.5

Properties of this stock (cured 20' at 320°F) are described as:

Tensile p.s.i. 1,075; Elongation, per cent, 290; 100 per cent modulus, p.s.i., 620; Shore 'A' Hardness 83; Crescent Tear p.i. 236.

This stock is stated to exhibit a high degree of resistance to common household agents (e.g. detergents, rinse aids, bleach etc.) which may cause severe deterioration of other rubber parts.

1959 IUPAC Conference at Munich

NEXT YEAR, the 20th conference of the International Union of Pure and Applied Chemistry (26-29 August) and the 17th international congress of pure and applied chemistry (30 August to 6 September) will be held in Munich. The scientific programme will cover inorganic chemistry (with a paper to be read by Professor H. J. Emeleus) and symposia on biochemistry and applied chemistry.

In the inorganic chemistry, sections will include: compounds with metal-carbon bonds; hydride chemistry. actinides and lanthanides; fluorine and fluorides; preparation of super-purity metals; water-like solvents; gas equilibria; semi-conductors; ternary oxides and sulphides.

The symposia will cover; natural pigments and their biogenesis; structure, biogenesis and synthesis of biologically important oligopeptides; possibilities of reactions under extreme conditions; treatment and utilisation of biogenic and industrial waste products; pest control.

Correspondence should be sent as follows: Congress: Meiserstrasse 1, Munich 2; conference: Dr. R. Mörf, secretary-general, IUPAC, Sandoz, SA, Basle 13, Switzerland.

New BOG Plant in Derby

A new factory for the manufacture of oxygen and dissolved acetylene has been opened by British Oxygen Gases Ltd. on the Raynesway Industrial Estate at Derby. The company now has 46 oxygen producing and compressing centres and 20 for dissolved acetylene in operation throughout the country.

Polarography Meeting In Scotland

Group of members attending the symposium

One-day Symposium Held at ICI's Nobel Division, Ardeer

A ONE-DAY Symposium on Polarography was held on 26 September in the Education Centre of Imperial Chemical Industries Ltd., Nobel Division, Ardeer, Ayrshire, by kind permission of ICI, Nobel Division. The meeting, which was attended by 50 members, consisted of morning and late-afternoon sessions for the presentation and discussion of eight papers on polarography, together with a visit to the Ardeer factory testing station in the early afternoon for a display of explosives in action. Members were the guests of ICI for lunch and dinner.

After registration at the Education Centre, delegates were welcomed by Dr. David Traill, research director, Nobel Division, ICI, Stevenston. The morning session, under the chairmanship of Mr. G. F. Reynolds, chairman, Polarographic Society, consisted of the presentation and discussion of the following papers: 'Polarography of sodium hydrosulphite and sodium formaldehyde sulphoxylate at the dropping mercury electrode' by Professor I. M. Kolthoff and Dr. N. Tamberg (University of Minnesota); 'Polarometric measurement of respiration in biological systems' by Dr. R. B. Beechey and Dr. F. Holton (Experimental Radiopathology Research Unit); 'The determination of conductivities using unconventional electrode systems' by Dr. V. S. Griffiths (Battersea College of Technology); 'Polarographic determination of dibutyl phthalate in propellant compositions containing nitroglycerine' by Mr. A. F. Williams (ICI, Nobel Division); 'An electrochemical separation process for use in polarography' by Dr. G. C. Barker and Mr. R. L. Faircloth (UKAEA, Harwell).

Chairman for the afternoon session was Dr. Traill, and during this session three papers were presented and discussed: 'A new electrode for cathode-ray polarography' by Mr. G. E. Penketh (ICI, Billingham); 'The polarographic determination of stability constants' by Dr. F. J. C. Rossotti (University of Edinburgh); 'Polarographic examination of the chelating power of EDTA and some closely related chelating agents' by Mr. W. Hoyle and Dr. T. S. West (University of Birmingham). The paper by Hoyle and West will be reported next week.

Polarography of Sodium Hydrosulphite and Sodium Formaldehyde Sulphoxylate

IN their paper on 'Polarography of sodium hydrosulphite and sodium formaldehyde sulphoxylate at the dropping mercury electrode', I. M. Kolthoff and N. Tamberg (University of Minnesota, US) review previous investigations. Thus reference is made to Furness[1] who reported on the polarography of sodium hydrosulphite and its impurities and proposed several procedures which find analytical applicability. His data on the diffusion current constant and the half-wave potentials of hydrosulphite were obtained in ammoniacal phosphate buffer (*p*H 10) containing 0.01 per cent gelatin as maximum suppressor. Kolthoff and Tamberg have found that gelatin decreases the diffusion current and affects the half-wave potential of the main anodic hydrosulphite wave at the dropping mercury electrode (DME), and also affects the characteristics of the hydrosulphite kinetic waves.

Cermak[2] and Peter[3] published results on the small anodic and cathodic kinetic currents observed in sodium hydrosulphite solutions, which are reported to be of equal height. In order to obtain an exact value of the diffusion current constant of hydrosulphite an accurate analysis of Kolthoff and Tamberg's commercial product was required under varying conditions of *p*H, temperature and concentration. To obtain the desired reproducibility of the analysis results, it was necessary to refine some of the methods published in the literature[4].

No quantitative studies on the polarographic behaviour of sodium formaldehyde sulphoxylate, $CH_2(OH)SO_2Na$ (denoted RSO_2Na), had been reported in the literature it was observed. Furness[1] had mentioned that RSO_2Na could be oxidised irreversibly at a stationary platinum electrode in a citrate buffer of *p*H 4, but that no anodic wave in this medium could occur at the dropping mercury electrode (DME). Kolthoff and Tamberg reported that RSO_2Na gives well-defined anodic waves at the DME in alkaline solutions (*p*H 9 to 13). Sodium formaldehyde sulphoxylate was reported to be stable in neutral and alkaline solution[6,7].

The average diffusion current constant of the anodic wave corresponding to the oxidation of hydrosulphite to sulphite was 3.82 ± 0.02 at 25°C. Gelatin, and to a lesser extent polyacrylamide (PAA), suppressed the diffusion current. In the absence of maximum suppressors, the half-wave potential was −0.50 volt at 25°C and became less negative in the presence of gelatin and PAA.

In neutral and alkaline solutions the ratio of the kinetic anodic prewave to the kinetic cathodic wave was 1.35. The cathodic wave was much more suppressed by gelatin than the anodic prewave.

Sodium formaldehyde sulphoxylate (SFA) yielded well-defined anodic waves in alkaline solutions (*p*H 9 to 13). The diffusion current constant was 3.84 ± 0.01 at 25°C. The half-wave potential was *p*H dependent. At *p*H 9, $E_{\frac{1}{4}}$ is -0.21_5 volt and became 0.06 v. more negative per unit increase of *p*H. The electrode reaction corresponded to the oxidation of the formaldehyde sulphoxylate anion to sulphite and free formaldehyde.

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5. PATEL, C. C., RAO, M. R. A. *Natl. Instit. o Science of India Proceedings VI*, 1953.
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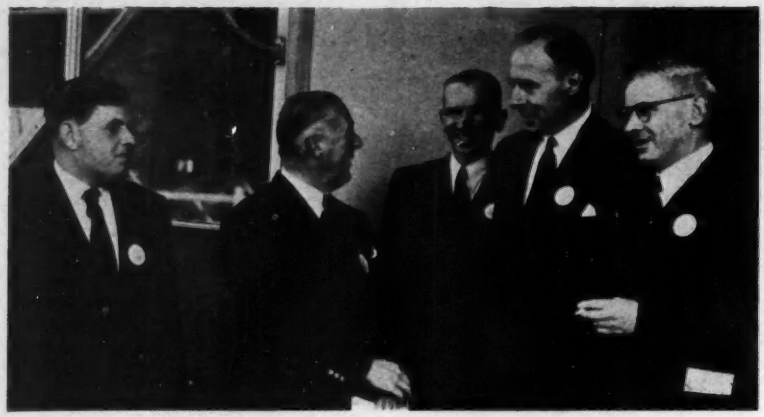
Discussion

Mr. G. Russell, Ilford Ltd., asked how accurate values of half-wave potential and diffusion current constant were obtained in the absence of maximum suppressors.

In the absence of the authors it was agreed that this point was not made clear in the summary. Mr. Williams added that the full paper would be published in the next *Journal of the Polarographic Society*.



Left to right: G. F. Reynolds, chairman, Polarographic Society; Dr. D. Traill, research director, ICI Nobel Division; A. F. Williams, Nobel Division research department; W. J. Parker, secretary, Polarographic Society; and R. C. Payn, Nobel Division research department

POLAROMETRIC MEASUREMENT OF RESPIRATION IN BIOLOGICAL SYSTEMS

DURING spectrophotometric investigations by Dr. R. B. Beechey and Dr. F. A. Holton (Medical Research Council, Experimental Radiopathology Research Unit, Hammersmith Hospital, London) into the nature of the mechanism of electron transport in the terminal stages of respiration, i.e., the reduction of oxygen to water, a requirement for an oxygen measuring device to work simultaneously with the spectrophotometer became obvious. The chosen method had to satisfy the following requirements: be capable of measuring changes in oxygen concentration in solutions (approximately 270 μM oxygen) to zero oxygen concentration; give rapid response to changes in rate of oxygen consumption; must not damage the respiring particles either mechanically or chemically; and the device had to be small for placing in a spectrophotometric cell of 2 mm. optical depth, and not interfere with the light beam.

In view of these requirements conventional manometric techniques were not suitable and a polarographic or amperometric method for the measurement of oxygen concentration was adopted. A dropping mercury electrode was excluded because the flow of the mercury would interfere with the light path and because mercury ions would inhibit and disrupt biological systems.

It was decided to use a vibrating platinum cathode, following the experience of Harris and Lindsay and also of Chance and Williams. Electrodes were constructed by placing platinum wire, diameter 0.009 in., in capillary tubes of lead glass, 4-6 cm. long and sealing the tube at one end. Approximately 0.8 cm. of polythene tube, of appropriate diameter, was fitted at the other end of the capillary and the protruding wire was then bent back over the polythene tube. A second piece of polythene tube was then placed over the first, so that the wire was trapped between them. The electrode could then be fitted to the vibrator by simply inserting the vibrating rod into the lumen of the second polythene tube. The actual lengths of wire acting as the electrode were gradually decreased during the investigation from approximately 10 mm. to 2 mm.

Experiments using yeast suspension illustrated that the cathode sensitivity (μamps per μmolar oxygen) was variable, not only from experiment to experiment but also from run to run within a single experiment. An important effect after exposure of the electrode to atmosphere and subsequent immersion was 'early artefact.' Results suggested that this phenomenon was the result of a slow approach to equilibrium around the electrode both after exposure and after disturbance of the diffusion layer on mixing.

In its present form the apparatus consists of a platinum electrode, approximately 2 mm. long, and mounted to be free of external vibration, inserted into a cell 1 × 0.5 × 4 cm. A saturated potassium chloride agar bridge leads from the cell to a calomel half-cell which serves as an anode. The electrode is cleaned with warm concentrated nitric acid before use and after this treatment gives a current of 6-12 μamps per μmolar oxygen. The early artefact lasts for a period of approximately half a minute on reimmersion after exposure and also on stirring the contents of the cell. The electrode is placed approximately 1 cm. above the base of the cell. Assuming that the vibration of the electrode does not create a stirring effect, it can be shown by calculation that at extremely low respiration rates atmospheric oxygen only diffuses to a depth of 1 cm.

Extensive use has been made of the apparatus to measure respiration rates and the effect of various compounds of the respiration of yeast cells and of rat heart sarcosomes (mitochondria) and of sarcosomal fragments. Rapid measurement of respiration rates is possible and combined with a pen-writing polarograph yields a clear record of the experimental results which can be quickly and readily assessed. Drs. Beechey and Holton claim that it has many advantages over the conventional manometric methods of measuring oxygen uptake and is an important addition to the spectrum of techniques available to the biochemist.

Difficulties of Removing Oxygen from Polarographic Solutions Discussed

Mr. G. E. Penketh, ICI, Billingham, stressed that the difficulties in removing oxygen had plagued polarographers for years, and asked if it was possible to remove oxygen from polarographic solutions with enzymes or similar materials.

Dr. Holton said that it would seem worth while to investigate whether respiring baker's yeast could be used for this purpose. From the work of Winzler (*J. of Cellular and Comparative Physiology* 1941, **17**, 263) it was known that a suspension of yeast would remove oxygen from solution at a constant rate until the oxygen concentration had fallen to about 10^{-4} Molar, and that a final oxygen concentration considerably below this value was finally attained. Whether the presence of the yeast would upset the subsequent polarographic determination would need to be studied explicitly for the solutions to be analysed.

Mr. R. Rooney, British Cast Iron Research Association, asked whether information could be given on the susceptibility of yeast to traces of metals and of the oxygen level in the anaerobic solution.

Dr. Holton replied that some indication of the relative intensity of intact yeast cells to metallic ions which inhibit isolated enzyme systems might be gained from considering the work of Winzler, who successfully studied the respiration of yeast cells with the dropping mercury electrode. In experiments lasting up to half an hour, there was no evidence of deterioration in the respiratory enzyme system. By contrast many isolated enzymes were very rapidly inactivated by mercury ions.

The oxygen level in a solution rendered anaerobic by yeast or animal enzyme-catalysed respiration was likely to be less than 10^{-7} Molar judging from the work of Winzler, Bänder and Kiese, and of Longmuir and his associates.

Mr. Rooney suggested the use of a

flow-through cell in which to wash the electrodes without exposure.

Dr. F. J. C. Rossotti, University of Edinburgh, added that in contemplating the removal of oxygen from solutions to be polarographed, by the addition of enzymes, it was necessary to consider the formation of enzyme complexes of metal ions already in solution.

Dr. Holton agreed that this could be significant if cell-free enzymes were added. The addition of yeast cells, i.e., whole cells with a selectively permeable cell wall, would probably prevent the immediate binding of trace metals with enzymes.

Mr. G. F. Reynolds said that the use of the polarograph was increasing in the fields of biological and medical research, particularly for oxygen determinations. He was himself working on a vibrating electrode in conjunction with Dr. T. S. Longmuir (Institute of Diseases of the Chest) and Mr. W. J. Parker (BPRI), which could be used to determine oxygen in the blood *in vivo*.

Electrochemical Separation Process for Polarography

A SIMPLE electrochemical method was described by G. C. Barker and R. L. Faircloth (AERE, Harwell) whereby metals which are soluble in mercury can be analysed polarographically for traces of more electronegative impurities. The technique was specifically developed for the analysis of bismuth using a square wave polarograph.

The technique consisted of adding a small volume of solution to a large volume of mercury in a small vessel and vigorously shaking the mixture for a short period. The more electropositive materials present in the solution tended to form a solution with the mercury, if they were soluble, while the more electronegative impurities would remain in the solution. By this means a considerable reduction in the concentration of major constituents could be achieved so that the impurities could then be determined in solution by normal methods.

The method had the advantage over constant-potential electrolysis of simplicity and economy.

In the case quoted, *viz.*, the determination of impurities in bismuth it was possible to remove 95 per cent of the bismuth in the solution and thus permit the direct polarographic determination of the impurities. Without this separation of the major constituent, it was not possible to carry out this determination since the excess of bismuth not only produced overlapping waves but also caused a marked turbulence in the mercury electrodes owing to the very large currents passing.

Mr. Reynolds said that this was a most important piece of work. One of the most difficult problems in the analysis of bismuth for trace elements was the initial separation from the major constituent. In the past he had used the method of a manganese dioxide 'collection' in which potassium permanganate was added to the sample solution and then precipitated as manganese dioxide. This carried down most of the important trace metals but left the bulk of the bismuth in solution. The method by Mr. Faircloth appeared to be much more elegant and predictable.

DETERMINING CONDUCTIVITIES WITH UNCONVENTIONAL ELECTRODE SYSTEMS

IN polarographic work it was often of value to have a knowledge of the conductivities of solutions under examination, but the use of conventional dipping electrodes for this purpose was accompanied by a number of experimental difficulties, said Dr. V. S. Griffiths (Battersea College of Technology). Many of these difficulties were associated with the electrodes and were often classified under the term 'polarisation', whilst in concentrated solutions corrosion and fouling problems were all too common.

Many attempts had been made to eliminate the use of immersed electrodes, mostly using high-frequency alternating current.

Using a three-terminal transformer ratio-arm bridge it was possible to determine conductivities with non-contacting electrodes as well as conventional electrodes and the frequency was of little importance, measurements having been made at 1,000 c/s, 1,592 c/s, and 10 Kc/s.

The cores of the voltage and current transformers could be linked by a closed annulus of conducting liquid contained in an insulating tube of glass or polythene. The transformers were moulded into an insulating material and where the electrolyte was inert with respect to the moulding the probe might be immersed in the test-solution, provided the container was large in comparison with the probe.

These types of unit had been used to determine the conductivities of a variety of solutions, being especially suited to concentrated solutions.

The electrolyte used varied from potassium ferro- and ferri-cyanides, through barium chloride to the conventional uni-valent electrolytes such as potassium bromide and hydrochloric acid.

The ease with which the bridge determines the capacitance term enabled it to be used as a 'dielectric bridge.' This application permitted the determination of impurities such as water in organic liquids.

Dr. R. J. Magee, Queens University, Belfast, asked whether the method would be suitable for the determination of small amounts of water in methanol where the dielectric is high since the method appeared to be dependent, for the determination of small amounts of water in organic solvents, on the pure solvent having a low dielectric constant.

Dr. Griffiths, in reply, said that the method was really at its most sensitive and most accurate condition in a dielectric constant range of 1—5 with a loss factor of 0—0.1. In this range it had been possible to determine low concentrations of water in cyclohexane as long as true solution existed. Outside this range little data was available, although acetone, of dielectric constant approximately 20 and having a relatively low loss factor, gives good results. It was anticipated that the alcohols would have a higher loss factor which might invalidate the method.

Mr. Reynolds believed that high frequency conductivity and dielectric constant methods had great applicability to industrial control of processes. In his laboratory, Mr. Leake was studying the determination of water in acetic acid by a continuous flow method based on similar principles. Mr. Reynolds also mentioned that recent gas chromatographic studies by Mr. D'Oyly-Watkins at Woolwich had revealed the surprising fact that acetic acid, water, and acid and hydride could co-exist for a considerable period in the same solution.

Dibutyl Phthalate in Propellant Compositions Containing Nitroglycerine

CHEMICAL procedure for the determination of dibutyl phthalate in propellant compositions (1) containing nitroglycerine was based on destruction of the nitroglycerine with ammonium sulphide followed by saponification of the dibutyl phthalate prior to titration of the phthalic acid. As this procedure was somewhat cumbersome for routine work, it was considered that a quicker and more direct method was desirable. Mr. A. F. Williams (Nobel Division, ICI) describing this new method said that dibutyl phthalate could be determined polarographically (2) using the normal polarographic method but in the presence of nitroglycerine there was marked interference, necessitating the introduction of an additional separation stage in the analysis.

Employing the cathode-ray polarograph it was possible to determine the dibutyl phthalate directly. Nitroglycerine caused an enhancement of the height of the peak obtained for the dibutyl phthalate but above a certain minimum value this effect was constant so that at the outset of an analysis it was only necessary to ensure that the amount of nitroglycerine present was exceeded. To determine dibutyl phthalate in a propellant mixture it was necessary to carry out an extraction with ether followed by the polarographic determination after removal of the ether. The base solution comprised 50 per cent methyl alcohol solution containing tetramethyl ammonium iodide. The method had an accuracy of ± 2 per cent, Mr. Williams reported.

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Discussion

Dr. F. A. Holton, Hammersmith Hospital, London, asked whether it was likely that the dibutyl phthalate which was added to the mixtures which were to be analysed was quite free from other organic phthalates. It was necessary to know this, since the polarographic method would be unlikely to distinguish between the different phthalates and, of course, different phthalates may behave differently in the propellant mixture.

In his reply, Mr. Williams said that the type of organic phthalate present in the mixture would affect the behaviour of the propellant. Since the propellants are made within fairly close limits some clue would surely be obtained from the figure obtained for the result, as to whether or not the particular phthalate had been added. Usually, of course, for routine control of the industrial process, the particular phthalate used would be known. Furthermore, the method would not distinguish between isomers such as iso and normal dibutyl phthalate.

Dr. R. J. Magee, Queens University, Belfast, wished to know whether a comparison had been made between results obtained using the cathode-ray polarograph and a method using chromatography which removes nitrocellulose, followed by conventional polarography. The question was raised because of the possibility that the nitrocellulose would affect the wave-height of the phthalate.

Mr. Williams pointed out that in the preliminary treatment of the propellant an ether extraction was carried out which removed only dibutyl phthalate, nitroglycerine, mineral jelly and ethyl centralite, so that the question of interference by nitrocellulose would not, of course, arise. It has been shown, however, that the method still applies in the presence of very small amounts of nitrocellulose.

Mr. G. Russell, Ilford Ltd., suggested that the enhancement effect due to nitroglycerine might be caused by adsorption of the nitroglycerine and this could possibly be tested by adding something which was known to be very strongly adsorbed.

Mr. Williams agreed that this was a distinct possibility and said that he would carry out a few experiments on the lines suggested by Mr. Russell. Mr. Williams asked *Mr. R. C. Rooney*, British Cast Iron Research Association, whether he had encountered any similar 'enhancing' phenomena on the cathode-ray polarograph. Mr. Rooney replied that in the case of the aluminium-solochrome violet complex different slopes were obtained for different dyestuffs concentrations. Mr. Rooney had ascribed this to viscosity effects but this explanation might not be correct. Mr. Rooney pointed out that he had also obtained some depressing effects on the cathode-ray trace.

Mr. G. L. Young, Cambridge Instrument Co., asked whether it was possible that the reduction products of nitroglycerine affected the wave-height of the phthalate ion. If not, could a controlled potential electrolysis method be used in order to remove the nitroglycerine?

Mr. Williams agreed that this was something that should be investigated but a further stage such as Mr. Young had suggested would, of course, lengthen the method and detract from its use in routine operation.

Mr. Reynolds expressed great interest in this work which seem to provide a method which avoided the tedious separation of nitroglycerin. It was hoped to try this in the Chemical Inspectorate. It seemed to him that there was also a good chance of determining a nitroglycerin itself, as well as dibutyl phthalate on routine basis, which would be highly desirable.

Polarographic Determination of Stability Constants

THE stability constant of a single mononuclear metal complex may be determined, according to standard texts on polarography, from the dependence of the half-wave potential of a dropping mercury electrode upon the free ligand concentration at the surface of the mercury droplets. However, in general, not one but a whole series of mononuclear complexes, consisting of ligands associated with central metal ions, will coexist in stepwise equilibrium with metal ions and ligands. This was stated by Dr. E. J. C. Rossotti (University of Edinburgh) in his paper on 'Polarographic determination of stability constants.'

Provided that the concentration of supporting electrolyte was sufficiently high so that the activity coefficients were effectively constant, the overall and stepwise stability constants could be defined. The average number of ligands bound to each central metal ion, and the fraction of free metal ions might also be defined and the stability constants determined from the experimental functions by a number of methods, including several physico-chemical techniques for systems of labile complexes.

The polarographic method was not rigorously applied to the determination of stepwise stability constants until some 30 years after the invention of the polarograph by Professor Heyrovsky. Provided that the diffusion of free metal ions is the rate determining process, the experimental functions may be determined from the shift in half-wave potential for reversible reduction of metal ions to a lower valency state, or to the metal. For the few metal ions which can be reversibly reduced the polarographic method has advantages over the use of metal electrodes, and the experimental technique is easier than using amalgam electrodes although the precision is lower.

A basically similar treatment is applicable to the unique case where the dropping mercury electrode is anodically oxidised to bivalent mercury complexes, but it is more direct and more precise to use a conventional mercury electrode.

For complexes which are reduced irreversibly the stability constants may often be determined polarographically by making use of competitive complex formation with other cations which are reduced reversibly at a more positive potential, and utilising the half-wave potential shifts.

An extremely simple competitive procedure is possible with very stable complexes of polydentate ligands.

Discussion

Dr. G. H. Nancollas, Glasgow University, stressed that in using the polarographic method for the determination of complexity constants it was necessary to ensure that reversible conditions prevailed, since when working with bivalent metal dicarboxylates, which were normally reversibly reduced, it was by no means certain that this was so, when determining the stability constants of the lower complexes.

In his reply, Dr. Rossotti said that the apparent irreversibility at low free ligand concentrations was probably due to the fact that the free ligand concentration at the surface of the mercury droplets could no longer be assumed to be equal to that in the bulk of the solution. The potential of the dropping electrode corresponded to higher free ligand concentrations as the proportion of reduced metal ion increased and the wave was drawn out.

Dr. R. J. Magee, Queens University, Belfast, noted that in the Hume-De Ford equation relating to the formation of mononuclear complexes it was necessary to ensure the reversibility of the redox system, an excess of complexing agent (the concentration of which should be much greater than the concentration of metal ions), and thirdly, that all equilibria were rapidly established, and asked what changes were necessary in the equation which the author had derived if condition three was not fulfilled. Dr. Magee added that since he was interested in the formation of a single mononuclear complex a plot of $E_{\frac{1}{2}}$ vs ln (ligand concentration) was used, and asked whether a straight line indicated the formation of a single complex, and the absence of a straight line the formation of more than one complex.

Dr. Rossotti replied that if it could not be assumed that the total ligand concentration equalled the free ligand concentration, then the latter could be obtained by the methods indicated in the last paragraph of the paper. If the system was not labile, more than one polarographic wave would result. The heights of the separate waves would be an exact measure of the concentrations of the separate complexes in very inert systems. In intermediate cases it was possible to obtain 'Apparent constants' which might be combined with independently obtained equilibrium constants to obtain the rates of association. Dr. Rossotti agreed with Dr. Magee's deductions from the plot and added that since,

$$\text{from equation (6), } d(E_{\frac{1}{2}})/d \log a = \frac{-RT\bar{n}}{zF},$$

a straight line would be obtained for such ranges of log(a) that $\bar{n}$ was a unique integer.

ICI Alkali To Take 50% More School Leavers

Lord Rochester has informed the Northwich and district local employment committee that ICI Alkali Division is prepared to increase the intake of boy school leavers by 50 per cent for seven years from 1960. He emphasised that the company was unlikely to need all this additional labour and it might not be possible to retain all the apprentices concerned on reaching journeyman status, but ICI recognised it was largely responsible for the local situation and was offering this contribution towards the solution of a serious problem

NEW MERCURY ELECTRODE FOR CATHODE-RAY POLAROGRAPHY

A NEW mercury electrode, surface of which is automatically renewed between potential sweeps, was detailed by G. E. Penketh (Research Department ICI (Heavy Organic Chemicals) Ltd.) The cathode consists of a mercury pool and the apparatus incorporates a timing circuit which opens a solenoid valve for a short pre-set time at the end of each potential sweep and allows mercury from a constant head supply to replenish the electrode.

Polarograms are stated to be similar in form to those obtained with the dropping electrode and are equally reproducible. The sensitivity is proportional to the electrode area, and by using a 12 mm. diameter pool an increase of some thirty-fold over the usual dropping electrode has been achieved.

Discussion on Paper by G. E. Penketh

Mr. R. Rooney, British Cast Iron Research Association, noted that with the new electrode no increase in the slope of the base line was obtained, whereas while working with the pool cathode an increase in slope had been obtained, and with a stationary pool, reproducible results were obtained with lead, cadmium, nickel, aluminium, solochrome and arsenic. Mr. Rooney inquired whether an agar bridge and independent anode had been used to reduce the cell size.

Mr. Penketh replied that the increase in sensitivity not only applied to the diffusion current but also to the residual current. Where the normal polarogram had a sloping base line the new electrode would reproduce this base line but a lower sensitivity setting. It was agreed that with a stationary pool reproducible results were obtainable for cadmium and lead over several successive sweeps without changing the surface. The present cell design was simple and convenient though it was possible that some advantage would ensue through using an independent anode.

Mr. W. J. Parker, British Polarographic Research Institute, referred to the vertical movement of the cathode mercury surface with changes in the applied potential, which Mr. Penketh had ascribed to attractive forces between the anode and cathode, and suggested that this movement with potential changes was an example of the electro-capillary effect, since the net force between the electrodes would act in a radial rather than an axial direction, and would therefore be unlikely to contribute towards the axial movement of the mercury. The electro-capillary nature of this movement could readily be shown by simple experiment since by applying a steadily increasing negative potential to the cathode and observing the direction of movement of the mercury it will be found that a reversal of the direction of movement occurs at the potential of the electro-capillary zero, approximately −0.56 volts in chloride media, whereas if forces due to the potential difference between electrodes were the causative factor, the cathode mercury would be expected to continue to move in the same direction with increase in potential difference. Mr. Parker suggested that the irreproducibility observed with indium might be associated with the degree of solubility of indium amalgam in mercury. For relatively insoluble amalgams it might be advantageous to replace the intermittent renewal of the mercury surface by one of the types of flowing mercury electrode such as that described in *Anal. Chim. Acta.*, 1956, **14**, 194. Such a unit would also have the merit of eliminating the need for mercury valves and the electrical equipment required to ensure synchronisation. Mr. Rooney added that although arsenic formed an insoluble amalgam, reproducible results were obtained for 15-20 sweeps using a mercury pool cathode.

In reply, Mr. Penketh said that indium was only one example from several which gave irreproducible results with a stationary pool.

Henry Wiggins £10 M. Plant Project

STEADY progress is reported on Henry Wiggins and Co. Ltd.'s £10 million modernisation and concentration scheme, now under way at Hereford. The project will mean the transfer of production from Birmingham and Glasgow over the next seven years and a planned expansion in output of 50 per cent.

It is claimed that the plant will be the largest and most modern plant for the production and processing of nickel alloy products in Europe. It will employ some 3,300, more than three times the present labour force. In due course some 500 workers at Glasgow will be affected where the works are due to be closed. Most of the 1,400 workers at Birmingham will also be affected but the already extensive laboratories there are being extended. Workers at both places are being encouraged to move.

The Hereford site of 52 acres was purchased in June 1956 from the Ministry of Supply. The plant at present occupies seven acres but half the site will be developed on completion of the present scheme.

Commercial Process for Hydrolysing and Ammoniating Calcium Metaphosphate

USE of calcium metaphosphate to make mixed granular fertilisers has been hindered by the fact that the reactors used could not tolerate a high enough liquid phase to result in an economic end product with a high, water-soluble P_2O_5 content. Researchers at Central Farmers' Fertiliser report that a new process, now in operation on a commercial scale unit by Illinois Farm Supply, overcomes this problem and yields a range of formulations.

In the process calcium metaphosphate alone or with normal superphosphate to supply some of the P_2O_5 is hydrolysed and ammoniated in a high concentration of sulphuric acid. A 1-ton per hour pilot plant using this compact, continuous, self-drying granulation process has been operated successfully for a year.

In the commercial plant, three pug mill reactors act as hydrolyser, ammoniator and granulator. The ammoniator liners and all internal moving parts are of type 20 stainless steel; the granulator is of mild steel.

Calcium metaphosphate (and other phosphate-containing compounds if used), sulphuric acid and water are fed into the hydrolyser. As the temperature of the reactants approaches equilibrium, nitrogen materials are fed into the ammoniator. Reactants from the ammoniator and granulator are discharged as a slurry, which passes to a liquids holding tank from whence it is recycled to the on-stream reactors. Water flow rates and other physical conditions are adjusted until the physical state of the slurry is considered right for granulation by addition of potash and product fines recycle.

The discharge of the granulator is taken from the slurry tank to a direct-fired concurrent rotary drier (time taken from start of feeding raw material to material in drier is stated to be 6 to 8 minutes).

The material from the drier is cooled and then screened. Small product (0.6 mesh) is returned to the granulator and large product (20 mesh) is crushed and used as formulations allow.

Jenista and co-workers of Central Farmers' Fertiliser indicate that assuming a delivered unit price of calcium metaphosphate equal to that of triple superphosphate, raw material economics are such that for a comparable degree of granulation and physical quality, the process is almost competitive with other processes that make high analysis materials. The process was patented last June.

1959 Essay Prize on Corrosion Science

THE education panel of the Corrosion Group, Society of Chemical Industry, is awarding a prize of to the value of £26 5s for an essay on any aspect of corrosion of metals and its prevention. Those taking part must be not older than 27 years on the closing date (31 March 1959). A length of about 4,000 words is suggested.

It is not essential to include results of original work; entries may consist of surveys of knowledge in a particular field, discussion of practical problems and suggestions for future developments in research, in application of knowledge or in organisation of corrosion-preventative measures.

Full details may be obtained from the Corrosion Group, SCI, 14 Belgrave Square, London SW1.

Glass Industry Seeks Silicones with Better Properties

ALTHOUGH the coating of glass bottles with silicones has proved of limited value so far, the discovery of a silicone or other material having the same lubricating effect but without the present complications would have many applications.

This was the general consensus of opinion expressed at a symposium recently held by the Society of Glass Technology, where speakers representing the suppliers of silicones, the glass industry, and users of glass containers discussed experiments that had been carried out.

The coating of glass bottles with silicones to reduce breakage, chipping and other damage was described as a complicated process with as many difficulties as potential advantages. It could be applied in the form of a solution or emulsion, either during or after manufacture and, in the case of multi-trip bottles, each time they were used. Treatment during the manufacture of containers was claimed to give a more durable coating, owing to a catalytic effect on the newly-made glass.

Although the literature on the subject published since 1954 had been extensive, it presented a very confused picture with disagreement on practically every point. Claims were often based on inadequate data and coloured by commercial considerations, and the widely-different methods of handling glass containers introduced other important factors.

Until more was known about the basic physics and chemistry of glass surfaces in the varying conditions of temperature, humidity and so on, a full understanding of surface treatment was impossible.

There was doubt as to whether silicones could resist the attack of caustic detergents as used on multi-trip bottles, and further investigation might be worth while. Surface treatment had been known to increase a bottle's bursting strength, and there was evidence that the chemical durability of glass was slightly improved although it had been found that siliconing actually hastened the weathering of glass.

Finally, silicone treatment was said to cause labelling difficulties, and it was too early to say whether special adhesives could solve this problem.

Pfizers Acquire Kemball, Bishops for Nearly £3 million

PFIZER LTD., of Folkestone, have purchased, for £2.9 million, virtually all the shares of the old-established fine chemicals firm of Kemball, Bishop and Co. Ltd., Crown Chemical Works, London, E3.

This, says Pfizer's chairman Mr. Richard C. Fenton, will establish the combined group in a strong manufacturing position in the fine chemicals field and strengthen the UK's position in the projected European free trade area.

Kemball, Bishop, who have a staff of about 650, are a private company founded in 1870. They manufacture a range of fine chemicals used principally in the pharmaceutical, food and beverage industries that have an expanding sale in Britain and overseas. In 1935 the company was granted a licence by Chas. Pfizer and Co. Inc., New York, for the manufacture of citric acid by fermentation processes, and Chas. Pfizer's became shareholders.

Pfizer Ltd., have substantial chemical manufacturing facilities at Sandwich, Kent, and are linked in research and commerce with the Pfizer network of companies throughout the world. All these resources will be available from now on to Kemball, Bishop.

The executive directors of Kemball, Bishop—Mr. W. W. Muir and Mr. R. F. Kemball, T.D., B.A., joint managing directors, and Mr. F. G. Hart, finance director—will continue on the board in their existing positions. The chairman of Pfizer's, Mr. Richard C. Fenton, joins the board as chairman, and Mr. P. V. Colebrook, managing director of Pfizer's, and Mr. B. Page, controller, have also become directors.

It is, however, the intention of Pfizer's that Kemball, Bishop and Co. shall continue as a separate entity handling the group's output of bulk fine chemicals.

New Durham Coking Plant

The first part of a new £4 million coking plant at Murton (Co. Durham) was formally inaugurated when 50 ovens were lit to start the drying-out process. By the end of November, the ovens will be ready for carbonising coal. When in full operation the ovens will carbonise 1,000 tons of coal daily, and produce 250,000 tons of high-grade metallurgical coke each year. Between 7½ million and 8 million cu. ft. of purified gas will be supplied daily to the Northern Gas Board and by-products will include concentrated ammoniacal liquor, tar, and 1 million gall. of crude benzol annually.

British Federation to Show at Kunstoffmesse

The British Plastics Federation is to exhibit in the International Plastics Fair, Dusseldorf (Kunstoffmesse 1959) 17 to 25 October 1959, in collaboration with the Board of Trade.

About 20 member-firms of the Federation will have exhibits on joint stands. An information bureau, covering all products of the British plastics industry, will be staffed by experts.

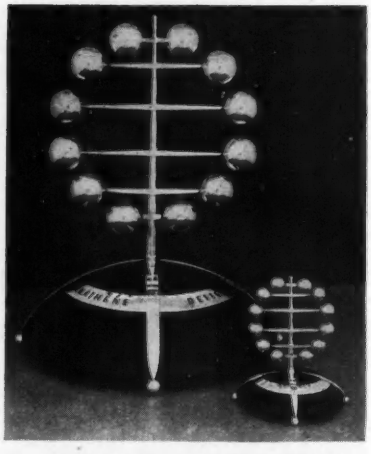
Argonarc Welded 'Dido' Tank at Harwell

THE DIDO reactor core and heavy-water reflector at Harwell are contained in an aluminium tank made by the APV Co. Ltd., Crawley. This is welded from 99.8 per cent pure aluminium by the British Oxygen Argonaut and Argonarc processes. It has a number of nozzles for coolant inlets and outlets as well as thimbles for experimental holes and the 'false-bottom' plenum chamber.

It represented a complex task since welding must be carried out to exacting standards, one of which was the necessity of identical analysis of the weld metal and the parent metal. There are three inlets for the heavy water leading to the manifold or plenum chamber into which the fuel elements are fitted. In addition, there are four outlets which take the form of bell-mouthed weir pipes situated at about the level of the outlet ports from the fuel elements, i.e. about 30 in. below the normal level of the heavy water, this upper layer acting as a reflector and having a considerable effect on the reactivity.

Heavy water entering the chamber at the bottom of the tank flows through the spaces between the fuel element plates, out of the fuel element ports and through the 7 in. weir pipes to a common header, feeding the pumps through Saunders valves.

Alkathene Design Trophy



For the first time, ICI Plastics Division are offering this year the Alkathene design trophy, to moulders using this polythene for retail articles, which have not been marketed before 1 January

Import Duty on Reforming Catalysts

The Board of Trade is considering an application to reduce the import duty on reforming catalysts consisting of platinum and/or palladium composited with an alumina or alumina and silica or silicate support, and containing, in addition to any sulphur, not more than 8 per cent by weight of fluorine and/or chlorine, and where the precious metal content is not less than 0.1 per cent and not more than 10 per cent by weight.

Overseas News

AMOCO'S NEW PHTHALIC ANHYDRIDE UNIT WILL PRODUCE 27,000 TONS A YEAR

NEW Joliet plant of the American Chemicals Corporation (Amoco) that will be commissioned in November for the production of phthalic anhydride and isomeric acids by a new process will have an annual rated capacity of 27,000 tons. The new process is said to allow for the production of both the anhydride and the acid by oxidation of 'a xylol-compound.' It can also be used for the oxidation of other aromatic hydrocarbons which are mineral oil by-products. It is hoped to put the process to other uses in the future, particularly in the development of acids at present without great commercial significance.

Annual phthalic anhydride capacities in the US are as follows: Allied Chemical Corporation, Barrett Division, 65,250 tons, National Aniline Division, 6,750 tons, Amoco, 27,000 tons, American Cyanamid, 33,750 tons; Koppers Co., 9,900 tons; Monsanti Chemical Co., 51,750 tons; Oronite Chemical Co., 30,600 tons (22,500 of which is isophthalic acid); Pittsburgh Coke and Chemical, 15,750 tons; Reichhold Chemicals, 18,000 tons; Sherwin-Williams, 3,150 tons; and Witco Chemical Corporation (under construction), 9,000 tons.

Market Secured For ANIC Fertiliser Output

Launching the new tanker *Agip-Gela* at Ancona, Mr. Enrico Mattei, president of ENI group to which ANIC's factory at Ravenna belongs, announced that 70 per cent of the fertilisers the factory plans to produce during the next eight years have been placed through Federconsorzi in various regions of Italy. The remaining 30 per cent will be exported.

There had been fears that ANIC would not be able to market the large quantity of fertilisers it has started to produce.

Du Pont To Build New Paint Plant In Belgium

A new plant to manufacture Du Pont paint products is to be built at Malines, Belgium. It will be operated by Du Pont de Nemours (Belgium) SA, a company to be formed soon as a subsidiary of E.I. du Pont de Nemours and Co., Inc. Output will include lacquers, enamels, paints, varnishes and thinners for the automotive, automotive refinish, industrial and household and maintenance fields.

The announcement was made by Mr. W. S. Carpenter, general manager of Du Pont's international department, at the Brussels International Exposition. Mr. Carpenter said: 'The decision was prompted by our strong faith in the future of the European economy and the wider trade opportunities under the common market treaty.' This will be Du Pont's first plant on the European continent since the war.

Mr. Stuart L. Finch of the parent company is going to Belgium to become production manager.

Design and construction will be handled by local firms, with building schdeuled to get under way in the beginning of 1959, and plant start-up in the autumn.

New Synthetic Fibres Plant Opened In Holland

A new plant for the production of Terlanka at Emmen, in Holland, was opened by Prince Bernhard of the Netherlands recently. The plant is on the premises of the factory of NV Enkalon, which has for some time been producing the fibre after which it is named at Emmen. The new fibre, Terlanka, is said to be particularly resilient, and will be used mainly, so the company says, in crease-resistant clothing, as filling for quilts and sleeping-bags and in the manufacture of certain types of curtains.

Shell Lift Phenol And IPA Output in US

Annual output of isopropyl alcohol and its derivatives by Shell Chemical Corporation has been increased by 120 million lb. following the expansion of production facilities at the Dominguez, Houston and Norco plants.

In February 1959, construction is to start on a new phenol unit at the Houston plant to meet the requirements of both Shell Chemicals and Shell Oil. Shell Chemicals will use the phenol in the manufacture of bisphenol-A, a starting material for epon resins, and Shell Oil will use it in the manufacture of lubricants and the recovery of aromatics. Acetone, used by Shell Chemicals as an intermediate in the production of other chemicals, will be a co-product.

Italians To Build Persian Plastics Plant

A plastics plant costing $6 million (£2 million) is to be built at Ahwaz, Persia, by Montecatini of Milan as part of the Persian seven-year plan.

West German Output and Import of Plastics Up

According to figures issued in Frankfurt-on-Main on 22 September, production of plastics in West Germany in the first half of this year reached 310 000 tons—an increase of 15 per cent. As in previous years, the greatest expansion has been in the field of polymerised products; in the six-monthly period these showed an increase of 18 per cent, at about 142,000 metric tons, on 1957 half-yearly figures.

Condensation products rose by 13 per cent (125,000 metric tons). Cellulose derivatives went up by 12 per cent to a half-annual total of 42,000 metric tons. A healthy expansion took place in the fields of phenol resin mouldings and glues, and in foams based on polyuretanes.

West German imports for the first half of 1958 increased by about 37 per cent, to a total value of DM80,600,000 (about £6,720,000). Exports went up by only 10 per cent, to DM255 million (about £21 million) in the period.

Styrene — Modified Polyester Withstands High Temperatures

Now available commercially from General Electric in the US are styrene-modified polyester resins. Compared with other polyesters, which withstand temperatures up to 300° to 400°F, these new resins are stated to withstand temperatures of 450°F and intermittent exposure at 500°F. With additional modifications, temperatures of 600° to 700°F can be withstood.

Experimental tests have shown that the modified resins will maintain flexural values in excess of 30,000 p.s.i., and compression values of about 20,000 p.s.i. after 200 hours' exposure at 425°F.

General Electric claim also that the products have better electrical specifications than glass-reinforced epoxy products.

The new resins can be 100 per cent polymerised and can be used in impregnation towers, it is claimed. The material can be fabricated by both vacuum bag techniques and matched metal die moulds.

Likely uses for these styrene-modified polyesters are radomes, aircraft ducting, commercial heat-ducting mechanisms and chemical tanks.

Reichhold To Establish Japanese Synthetic Resin Company

Plans have been announced by Reichhold Chemicals Inc, US, of the establishment of a joint company with Hong Kong industrialists of a Reichhold affiliate for the manufacture of the company's entire group of synthetic resins. The affiliate, which will be known as Reichhold Chemicals (Hong Kong) Ltd., will build a plant in Hong Kong to be ready for production by the spring of 1959.

East German Carbide Plant Plans To Be World's Biggest

The East German VEB Chemische Werke Buna plan to become the world's largest single producer of carbide. The company is to receive substantial financial grants for expansion, much of the money coming from the USSR.

These investments to be made during 1959, will be allocated mainly to the company's carbide production plant. The National Planning Commission claims

that by 1965 the plant will be turning out 800,000 metric tons of carbide annually.

Nine-tenths of the company's total products are based on carbide.

NBS Evaluation Of Leather Impregnants

Experiments conducted for the US Navy by the National Bureau of Standards have shown that a leather impregnant must be deposited throughout the leather's thickness in order to be effective in improving wear and water resistance. Best results, as well as ease of handling, are achieved, it is reported, when the elastomer components (such as polybutene, polyisobutylene, or butyl rubber) having a molecular weight of less than 10,000 are used in the impregnating solution. Depositions of approximately 20 per cent of the low-molecular-weight materials in the leather increase wear as shown in actual service tests 50 per cent and reduce water absorption 35 per cent.

US Celanese Plan To Expand Output Of Acetic Acid

An immediate start of a multi-million dollar expansion of manufacturing facilities for acetic acid is planned by Celanese Corporation of America. The expansion will double production capacity at the company's Pampa, Texas, plant, it is stated. Work is expected to be completed late in 1959.

Austro-Italian Petrochemical Company

Documents setting up the joint Austro-Italian petrochemical concern Danubia Petrochemie AG, have now been signed in Vienna by representatives of the nationalised Oesterreichishe Stikstoffwerke and the Italian Montecatini trust. The Danubia plant is expected to be completed by the end of 1960 and will be designed to produce 5,000 metric tons of polypropylene annually.

Plant Protection Chemical Sales in Finland in 1957

According to the Inspector of Plant Protection Service in Finland, Mr S. Roivainen, there were 21 manufacturing and importing companies of plant protection chemicals. Of the total of 230 such chemicals on the market, 41 were fungicides, 74 agricultural pesticides, 81 household, store-room and cowshed pesticides, 22 weedkillers and 13 preparations used as fungicides and pesticides.

Mercuric seed-dressing preparations' sales totalled 169 tons, which amount is stated as being sufficient for seed dressing of almost 50 per cent of seed used in 1957. Consumption of other mercuric preparations was 8.2 tons; that of copper sulphate and copper oxides, 3.4 tons (11.0 tons in 1956); of lime sulphur, 22.7 tons (30 tons in 1956); of captane + TMTD, 10.5 tons; of PCNB dust, 4.5 tons (10.5 tons in 1956); and of TCNB + 1PC, 10.5 tons (16.9 tons in 1956).

Agricultural pesticides are reported as having contained a total of 9.6 tons parathion (8.6 tons in 1956), 1.0 tons malathion, 5.8 tons of DDT, 0.6 tons lindane and 6.7 tons potassium and sodium arsenate. Sales of agricultural dusts reached 272 (337 tons in 1956). Consumption of petroleum and tar-oil winter washes totalled 97 tons (112 tons in 1956) and that of DNOC winter washes 10.0 tons (20.3 tons in 1956).

Household, store-room and cowshed pesticides contained 0.32 tons of parathion, 5.3 tons of DDT, 1.4 tons of lindane and 0.31 tons of pyrethrin. Rodenticides took 121 kg. of dicoumarin and 99 kg. of tallium sulphate.

Sales of weedkillers showed a small increase. Consumption of MCPA sprays was 284 tons and that of MCPA wettable powders 9.8 tons; of 2,4-D + 2,4,5-T-sprays 45 tons (41 tons in 1956); of DNOC and DNBP 2.3 tons; of aromatic oils 8.0 tons; of chlorinated acetic-acid preparations 30 tons (47 tons in 1956), and of non-selective weedkillers 108 tons.

USSR Produced 250,000 Tons Of Plastics In 1957

According to the Russian Ministry for Chemical Production, the following metric tonnages of plastics were produced in USSR last year: phenol resins, 64,800; urea and melamine resins, 53,800; epoxy and alkyd resins, 28,200; fluorine-content and similar materials, 20,000; caprolactum nylon, 12,800; silicone, 2,200; and assorted thermoplastics, 60,500. These represent an annual total of 242,300 metric tons to be increased eight-fold by 1965, according to Mr. Kruschev in a speech made earlier in the year.

German Chemical Apparatus Manufacturers Quiet Year

Figures released in Frankfurt at the end of last month show that the increase of production of chemical apparatus fell sharply in Federal Germany during last year. The percentage increase over 1956 was one of 4½ per cent; this compares with an increase in 1956 over 1955 of 35 per cent.

Exports were higher than in 1956 by 46 per cent. However, this figure too was down on the 1956 increase on 1955 —one of 50 per cent.

Determination of Acetylene in Low Concentration for Pollution Control

A RAPID, accurate, colorimetric method for detecting and measuring minute quantities of acetylene (as low as 10 p.p.m.) in air has been developed by E. E. Hughes and R. Gordon, Jr., of the National Bureau of Standards' gas analysis laboratory in co-operation with the US Public Health Service[1]. Since acetylene in the air is associated with the contaminants from automobile exhaust gases, the method is expected to be useful in the study and monitoring of urban air pollution. The equipment required is simple and portable, so that the method is well suited for field studies.

The first step in the procedure is concentration of a measurable amount of the dispersed acetylene by adsorption on silica gel. The quantity of acetylene present is indicated by the depth of colour produced when the silica gel surface is treated with a solution of ammoniacal cuprous chloride. Exact concentration is determined by visually comparing this colour with those produced by gas samples containing known concentrations of acetylene.

To begin the determination, a definite volume of air contaminated with the acetylene is drawn through a long, small-diameter glass tube containing highly purified silica gel the tube being cooled to the temperature of dry ice. Acetylene is adsorbed on the gel's surface. A column of fine glass beads bounds the gel layer at each end of the tube, making possible easier detection of slight colour changes. As acetylene collects mainly on the initial portion of the gel, the tube should be allowed to warm to room temperature for a short time to obtain a homogeneous distribution. An ammoniacal solution of cuprous chloride—obtained by mixing a solution of ammoniacal cupric chloride with hydroxylamine hydrochloride—is then introduced into the tube and the copper acetylide formed precipitates directly on the gel surface. The resulting colour ranges from pink to brown, depending on the quantity of acetylene.

When the concentration of acetylene in the atmosphere is 10 p.p.m. or above, detectable quantities can be adsorbed at room temperature. At concentrations below 10 p.p.m., the gel must be cooled to −78°C. At this temperature, Hughes and Gordon report, it has been possible to detect concentrations as low as 10 parts per billion.

The concentration of acetylene in the unknown air sample is determined by comparing the colour of the unknown with that of a tube exposed to a sample of known concentration.

Acetylene collected in the field can either be treated with the reagent immediately, or stored at a reduced temperature in liquid nitrogen and then returned to the laboratory for analysis. Collected acetylene which has been treated with the cuprous chloride solution can be stored for at least 48 hours without apparent loss of colour.

Experiments have indicated that this method can identify total adsorbed quantities of acetylene as low as 0.01 μg. contained in 12 ml. of air at a volume concentration of 1 p.p.m. Differences of 0.012 μg. between specimens of acetylene were easily observed in the range of 0.00 to 0.237 μg.

It is suggested that besides its use in the study of air pollution, this method can be applied to other industrial or laboratory processes where determination and fractionation of air constituents are required, as for example, determining the accumulation of solid acetylene in liquid air generators.

Analytical Review

FLUORINE ANALYSIS BY PYROHYDROLYTIC SEPARATION

By

T. S. West, Ph.D.

Dr. West reviews some recent developments in methods for the separation of fluorine from rare earths, thorium, aluminium, etc., by the technique known as pyrohydrolysis. These methods appear to have a wider range of application and to be more rapid than the fluosilicic acid distillation method. The review concludes by noting a new development in the use of acid-base ion-exchange indicator resins in titrimetric analysis.

MUCH interest is currently being shown in American laboratories in the analysis of fluorine, particularly in rare earths, etc., by the technique known as 'pyrohydrolysis'. The term 'pyrohydrolysis' used here has a somewhat different meaning from that attributed to it by Feigl[1] who was concerned with the hydrolysis of organic compounds using 'dry water' sources such as oxalic acid and manganous sulphate. In the present instance the word 'pyrohydrolysis' was coined by Wharf, Clive and Tevebaugh (1954)[2] and describes the decomposition of inorganic fluorides by heating them strongly (bright red heat) in a platinum tube while passing steam over the sample. The hydrofluoric acid thus formed is titrated with standard alkali.

The method is based on earlier work reported by Prideaux and Roper[3] in the *Journal of the Chemical Society* (1926). Wharf, Clive and Tevebaugh recognised the potentialities of the method and extended its use to the analysis of many metal halides. Fluorides were divided into two groups:—those easily hydrolysed, e.g. Al, Mg, Zn, Bi, Th and U and those difficult to hydrolyse, e.g. Be, Ca, Ba, Sr, Na and K. Banks, Burke and O'Laughlin[4] report work on the analysis of rare earth fluorides by pyrohydrolysis in a recent issue of *Analytica Chimica Acta*. It was shown by these authors that it is possible to determine fluorine in rare earths accurately and, in some instances, rapidly by this technique.

Lengthy Hydrolysis for Basic Rare Earths

Lanthanum and the more basic rare earths require excessive time for completion of hydrolysis and demand the use of an accelerator such as U_3O_8 and/or Cr_2O_3. These accelerators decrease the pyrohydrolysis time by as much as 50 per cent and give a precision comparable with that of unaccelerated pyrohydrolysis of a rapidly decomposed compound. Lutetium, ytterbium, cerium, scandium, gadolinium, terbium, dysprosium, holmium, erbium and thulium fluorides can be pyrohydrolysed within 30 minutes whereas ytterium, lanthanum, praseodymium, neodymium, samarium and europium fluorides require from 45-150 minutes. The use of an appropriate accelerator reduces the decomposition time to 30 minutes or less for all these fluorides except those of lanthanum, praseodymium and neodymium.

The platinum ware necessary for this work is expensive and not surprisingly several attempts have been made to reduce the costs. Thus Susano, White and Lee[5] suggested the use of nickel apparatus in place of platinum while Gillies, Keen, Lister and Rees[6] employed a silica apparatus. One of the most recent developments replaces the superheated steam used by earlier workers with a stream of moist oxygen[7]. Under these conditions tungstic oxide was found to be a more effective accelerator than uranium oxide at a lower temperature. The apparatus used was of silica construction throughout. This most recent development is of interest in several ways. The authors point out for example that it is a pyrolytic rather than a pyrohydrolytic procedure. It is claimed to be generally more rapid than either the Willard and Winter distillation technique[8] or the pyrohydrolytic procedure. The hydrofluoric acid is collected in a much smaller volume of solution thus ensuring more favourable end-point conditions.

A systematic study of effects produced by varying operating conditions revealed that the rate of recovery of fluorine increases with rise of temperature both at the reaction site and at the exit end of the tube. The flow rate of the oxygen was also quite critical. Thus at 80 litres per hour 1 meq. of fluorine was recovered in 15 minutes whereas at 100 litres per hour this was achieved within one minute's time. Best results were obtained with oxygen saturated with moisture at room temperature. The function of the water appears to be largely the replacement of fluoride adsorbed on the walls of the silica combustion tube.

The method is fairly precise even for microgram amounts. Thus 10γ of fluorine was recovered with a coefficient of variation of 5 per cent when sodium fluoride was used as the source with uranium oxide as accelerator. On the other hand the coefficient was only 0.1 per cent with amounts of the order of 4 meq. whether uranium oxide or tungstic oxide was used as accelerator. No bias was observed with tungstic oxide but a tendency to low recoveries was marked with uranium oxide. Phosphate and silicate do not interfere but sulphate, chloride and nitrate yield high results when a titrimetric finish is used. If, however, a spectrophotometric titration with thorium and Alizarin Red S (or Thorin) as indicator is applied, no interference is observed.

There is a need for a rapid reliable method for the determination of fluoride in compounds of thorium, uranium, rare earths, etc., and it may well be that these procedures based on pyrohydrolytic separation will prove to be satisfactory. It should be noted in conclusion that these methods are applicable to a wider variety of materials than the hydrofluosilicic acid distillation method of Willard and Winter. Al, Zr and gelatinous SiO_2 which retard or prevent liberation of fluoride by distillation offer no difficulties in these newer procedures.

Acid-base indicators which are themselves weak acids or bases may readily be adsorbed on strong anion or cation exchangers respectively thus imparting acid-base indicator properties to the resin[9,10]. Phenolphthalein, thymol blue, bromocresol green, etc., are readily adsorbed on anion resins such as Amberlite IRA 400 which are virtually colourless themselves. Thus a colour change at the end-point of an acid-base titration is readily seen when a few beads of the indicator form of the resin are added to the titration vessel. The colour change of the different indicator beads at the end-point is instantaneous whereas conversion of the resin to the indicator form is somewhat slow. Indicator resin beads may be used several times over after washing with distilled water and they may be dried and stored for future use.

Indicators chemically Linked to Anion Resins

Evidence is presented by Miller[9] to show that indicators such as phenolphthalein are linked chemically to anion resins and not merely adsorbed whereas the latter state of affairs appears to hold for cation exchange indicator resins. Indicator resins prepared from methyl orange, methyl red and congo red are not very successful. As with ordinary titrations it is sometimes advisable to conduct an indicator blank.

The main advantage of this technique seems to be the pin-point concentration of the indicator which is achieved because of the ability of the resin beads to load up to exchange capacity with the indicator. As little as one resin bead may be used when desired.

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Cyclopentanone, Versatile Starting Material, Now Available from Price's

PRODUCTION on a commercial scale of cyclopentanone, a versatile starting material of potential interest for a wide variety of applications, has been developed by Price's (Bromborough) Ltd., manufacturers of oleochemical products, Bromborough Pool, New Ferry, near Birkenhead, Cheshire.

Cyclopentanone (ketopentamethylene or ketocyclopentane) is a saturated cyclic monoketone, which occurs naturally in light wood-tar oils. It is offered to the following specification: Assay 99 per cent; acid value (mg. KOH/g.) 0.4 max.; specific gravity at 15°C 0.946/0.948; refractive index at 25°C 1.4344/1.4347; colour (Lovibond units 5¼ in. cell) 1.5 yellow max., 0.7 red max.; and boiling range 129/131°C. While it is of interest as a general solvent, cyclopentanone has been found to have special solvent properties for several types of polymer, particularly p.v.c.

One of the main applications for cyclopentanone is believed to lie in the field of synthetic perfumery and flavouring materials. Conversion of cyclopentanone to α-chloro-cyclopentanone, followed by dehydrogenation gives the unsaturated ketone cyclopent-2-en-1-one, the parent compound of the jasmone and pyrethrone series. A number of potential perfumery synthetics can be obtained from cyclopentanone. Suggested reactions are: Enol esters prepared by reaction with the anhydrides of monocarboxylic acids; reduction to cyclopentanol followed by esterification with aliphatic and aromatic carboxylic acids; alkylidene and arylidene cyclopentanones formed by condensation with various carbonyl compounds particularly aldehydes; ionone analogues by condensation with citral; and ketals by condensation with glycerol and glycols.

The semicarbazone has been claimed as a useful insecticide and as a minor component of an insecticidal aerosol. Use of cyclopentanone as a textile auxiliary (as shrinking agent for stretched cellulose acetate rayon and when sulphonated by action of chlorosulphonic acid, as a yellow/brown acid dye for wools) has been noted.

Other suggested uses of cyclopentanone listed by Price's (Bromborough) are: (i) Oxidation of cyclopentanone with nitric acid gives glutaric acid. A hydroperoxide is obtained on oxidation with hydrogen peroxide which may be of some interest as a polymerisation initiator (US Patent 2,298,405).

(ii) Reduction of cyclopentanone gives cyclopentanol used for making lubricating oil additives (US Patent 2,456,692).

(iii) Reaction with liquid ammonia gives a diamine derivative for which certain fungicidal properties have been claimed, as well as possible uses as an antioxidant and oxygen-scavenger (US Patent 2,693,491).

(iv) Condensation with acetic acid gives a product which can be used as a satisfying drying oil (German Patent 84,815).

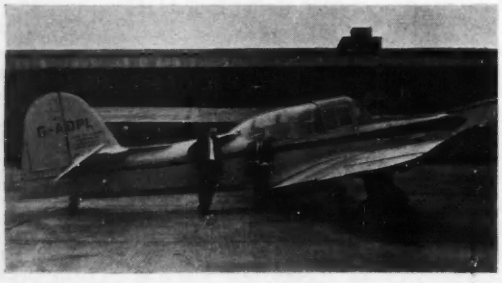
Two Balfour Group Salesmen Leave for Air Tour of Africa

TWO young sales engineers of the Balfour Group of Companies, Leven, Scotland, have set out in a single-engined four-seater Percival Prentice aircraft on a 22,000 miles tour of Africa to seek new customers for the firm's chemical process plants.

They are Ronald J. Hannaford, former RAF bomber pilot, who will be at the controls, and Gordon Pearce, who will navigate. Both have been with the firm for five years and know Africa and its sales potential well.

The route planned is via British West Africa across to British East Africa, through Rhodesia and Nyasaland and on to the Union and South West Africa, touching down at 41 potential 'selling points' on the way.

Although the flying salesmen are equipped with much technical data specially tabulated for the trip, enquiries for large projects will be dealt with by specialists who will fly out from Scotland with additional information for on-the-spot discussions.



R. J. Hannaford and G. Pearce before take-off from Southend Airport on their 11-week sales mission to sell equipment and services of the Balfour Group, Leven, Scotland

DIARY DATES

MONDAY, 13 OCTOBER
SCI—London: Royal Society of Medicine (and Tuesday), Symposium, 'Texture in food'.
RIC—London: Ewell County Technical College, 6.30 p.m. 'The chemistry and application of beryllium', by Dr. T. W. Farthing, M.A., Ph.D.

TUESDAY, 14 OCTOBER
SCI—London: 14 Belgrave Square, S.W.1., 6 p.m. 'Titanium and its alloys as materials of construction for chemical plant', by Dr. K. W. J. Bowen.
SAC—Nottingham: Gas Showrooms, 7 p.m. 'Identification of the new permitted food colours' by P. S. Hall, F.R.I.C.

WEDNESDAY, 15 OCTOBER
SAC—London: 'The Feathers', Tudor St. E.C.4., 6.30 p.m. 'The role of the microchemist in industry' by C. Whalley, F.R.I.C. and E. G. Ingram.
Plastics Inst.—Leicester: Grand Hotel, 6.45 p.m. 'Polypropylene' by Dr. F. Ranalli and Ing. G. Crespi (Montecatini SPA).
SAC—Glasgow: Kenilworth Hotel, Queen St., C1, 7.15 p.m. 'Acidity in dark lubricating oils' by Dr. W. Gibb and H. Gibson, B.Sc., and 'Volumetric analysis of stannous and total tin in soluble tin compounds' by J. D. Donaldson B.Sc., and W. Moser, B.Sc.

THURSDAY, 16 OCTOBER
SCI—Liverpool: Nicholson Theatre, The University, 7.30 p.m. 'Organic chemistry—the changing scene' by Sir Ian Heilbron.
CS—London: Imperial College, South Kensington, S.W.7., 7.30 p.m. Centenary lecture 'Spectra of free radicals' by Dr. G. Herzberg, F.R.S.

FRIDAY, 17 OCTOBER
Plastics Inst.—Birmingham: James Watt Memorial Inst., Gt. Charles St., 6.30 p.m. 'Polypropylene.'
SAC—London: Courtauld Hall, Queen Elizabeth College, University of London, 2.30 p.m. 'Spectropolarimetry' by Dr. M. K. Hargreaves, Dr. W. Klyne and Mr. A. C. Parker.

Market Reports

Buyers Still Cautious

LONDON Trading conditions have again shown little change with buyers on home account continuing to follow a cautious policy. Nevertheless, the routine industrial chemicals are moving steadily against contracts and the flow of export inquiry has been on a good scale although keen quotations are being sought.

Sulphate of copper is moving reasonably well on export account at the higher price now operating. Borax, boric acid and hydrogen peroxide are reported to be in good demand.

MANCHESTER Replacement buying on the Manchester chemical market during the past week has again been less active than had been the experience until a relatively short time ago, most consumers being content to cover their needs over shorter periods. In most instances, however, they are specifying against commitments fairly regularly.

GLASGOW The past week showed some improvement on the Scottish heavy chemical market with an overall increase in activity, particularly towards the end of the week. Demands were varied, and quantities were well maintained, showing, in some cases, an upward tendency. Some slight variation in prices did take place, but mostly they remained on a firm basis. There is little change in regard to agricultural chemicals, the seasonal quietness still prevailing. A little more interest was shown in exports.

Commercial News

- **Allen & Hanburys Profits up £49,000**
- **Glaxo Report £772,000 Rise in Profits**
- **Coopers Sell Plant Protection Shares**
- **Milton Recommend Acceptance of Vick Offer**

Allen and Hanburys

Allen and Hanburys report that group net profits increased from £152,775 to £201,723 in the year to 30 June after tax of £236,389 (£193,672).

The final dividend is repeated at 10 per cent on the £488,400 ordinary capital to make 17½ per cent for the year. This is as previously when the interim was on a smaller capital. The parent's net profit is £153,886, compared with £111,347.

Ashe Chemical Ltd.

An interim dividend for the year ending 31 December 1958 of 5 per cent, being 0.6d. per share, less income tax, on the ordinary shares of 1s each has been declared by Ashe Chemical Ltd.

Glaxo Laboratories

A rise of £772,566 in group net profits, a 5 per cent increase in the dividend for the year ended 30 June 1958 and a one-for-two scrip issue are announced by Glaxo Laboratories. Net profit, after all charges, including £3,148,000 (£2,332,000) for tax, is £2,610,566 against £1,838,000.

With the rise in the interim dividend from 5 per cent to 6¼ per cent, the final dividend is higher at 11¼ per cent (7½ per cent). This is on increased capital as a result of the acquisition of Allen and Hanburys.

The proposed scrip issue will involve capitalisation of £2,755,350 of which £2,014,650 will come from the share premium account arising on the acquisition of the ordinary share of Allen and Hanburys. The balance of £740,000 will be from the general capital reserve.

Glaxo and Allen and Hanburys have agreed on proposals for reorganising their preference share capital. To effect the scheme, Glaxo will create a new class of 6 per cent cumulative preference stock. If the proposals are approved, holders of each class of preference and preferred ordinary capital in both companies will receive an increase of 10 per cent gross in the amount of annual income.

ICI-Cooper McDougall

Cooper McDougall and Robertson have sold their holding of £500,000, 13 per cent cumulative preference shares in Plant Protection Ltd. to Imperial Chemical Industries Ltd., for £1 million. Under the agreement, the proceeds have been used to subscribe for £1 million new 5 per cent cumulative preference stock of ICI. The agreement also provides for maintenance of the income hitherto enjoyed by Cooper McDougall from the preference shares in Plant Protection.

Milton Antiseptic

The formal offer of 34s 1½d. per 10s share for the £196,152 ordinary capital of Milton Antiseptic has now been received from Vick International, the English subsidiary of Vick Chemical, of the US. The Milton directors who own or control 89,670 shares and hold 38,566 shares as trustees, propose to accept in respect of their shares, and recommend acceptance by other holders.

Vick, it is reported, desire to extend their operations in the pharmaceutical field, but not to include such products as are manufactured and sold by Milton's Deosan division. Vick therefore propose that subject to the offer becoming unconditional, Milton should sell for cash the Deosan division to Diversey (UK) recently incorporated and held by Diversey Corporation of Chicago.

Milton chairman is Mr. E. J. Tucker. On the present offer becoming unconditional, Mr. E. R. Alltree and Mr. W. Stannard will retire from the Milton board and the remaining directors will continue in office.

Lawes Chemical Co.

Lawes Chemical Co. Ltd., although showing a lower profit for the year ended 30 June last, had a satisfactory trading year during which turnover was increased. The company's share of the national increase in fertiliser sales was also maintained.

At the annual meeting this week, the company's chairman, Mr. F. A. Perkins, announced that production had been resumed at the main plant at Barking, Essex. The plant had been modified and adapted to steam granulation. Combined with the 'High Dry' process already in use, the Barking plant could now give increased production with better quality both from the sowing and keeping point. Merchants and farmers would be able to store Lawes fertilisers during the winter, it is claimed.

Prices for fertilisers for the season 1958/59 have been based on current costs of materials and freights, enabling the company to pass on to their customers reduced prices.

The board feel that a satisfactory position is shown in the company's accounts and payment of 14 per cent dividend for the year to 30 June 1958 is recommended.

Morgan Crucible Co.

Morgan Crucible Co. have disposed of their financial interests in Carblox to Thomas Marshall and Co. (Loxley) who assume complete control.

Tube Investments

Group net profits of Tube Investment for the year ended 31 July 1958 were nearly £500,000 higher at £6,003,438 compared with £5,510,909. Trading profits of the subsidiaries expanded from £15,650,685 to £16,104,804. Taxation took £7,032,394 as against £6,635,320. Depreciation is put at £3,525,560 (£3,676,402).

Final dividend is raised from 7½ to 10 per cent to make 17½ per cent for the year, as against 15 per cent for 1957. The dividend takes £1,484,645 against £1,258,568.

Chemische Werke Hüls

Chemische Werke Hüls AG, of Marl, near Recklinghausen, W. Germany, have declared a dividend of 10 per cent as against 8 per cent last year. Turnover has risen, both at home and abroad, by 9.6 per cent on 1956 totals.

Turnover of home sales, says the annual report, rose from DM 306 million (about £25,300,000) to DM 338,800,000 (about £29 million) and that of overseas sales from the 1956 total of DM 171,400,000 (about £14,700,000) to DM 184,300,000 (about £15,800,000). Exports proportion remained constant for 1957 at 35 per cent, in spite of increased competition from North American companies operating in Europe and Japanese companies in Asia. Competition was particularly keen in the plastics field and many new products had come on to the market. Import restrictions by the French Government during the financial year had added to the difficulties.

Capital expenditure of about DM 64,100,000 (about £5,350,000) was invested during the year, DM 16,100,000 (about £1,350,000) going on the setting-up, with Badische Anilin- und Sodafabrik AG, Farbenfabriken Bayer AG and Farbwerke Hoechst AG, of the Bunawerke Hüls GmbH on Chemische Werke's premises. Chemische Werke Hüls will raise their share capital by DM 60 million (about £5 million) by 1 June 1963, in two stages. The number of employees of the firm rose from 13,271 to 13,957 in the year under review.

Reichhold Chemie AG

In offering another DM 1 million (about £83,300) shares to the public, the Hamburg firm of Reichhold Chemie AG announce an annual turnover of more than DM 25 million (about £2,080,000), about 5 per cent of the world turnover of the Reichhold group owned by Reichholds Chemicals Inc., of White Plains, NY. The company's dividend for 1957 was 11 per cent.

Shawinigan Water

Dividend of 17 cents a share on the no par value common shares of Shawinigan Water and Power Co. for the quarter ending 30 September 1958, has been declared. A dividend of 33¼ cents a share has also been declared on the company's A common shares.

INCREASES OF CAPITAL

ROUSSEL LABORATORIES LTD, chemical manufacturers, etc., 847 Harrow Road, London NW10. Increased by £100,000 beyond the registered capital of £200,000.

CYCLO CHEMICALS LTD., 376 Strand, London WC2. Increased by £10,000 beyond the registered capital of £10,000.

UPJOHN OF ENGLAND LTD., manufacturers of chemicals, pharmaceuticals, etc., Fleming Way, Crawley, Sussex. Increased by £175,000 beyond the registered capital of £35,000.

NEW COMPANIES

EVANS MEDICAL (MIDLANDS) LTD. Cap. £100. Manufacturing chemists. Directors: I. V. L. Ferguson, W. A. Kinnear, C. W. Robinson and G. A. Wilson. Reg. office: Speke, Liverpool 24.

UNIBRIX RADIATION SHIELDINGS LTD. Cap. £5,000. Manufacturers of and dealers in bricks, radiation shielding materials, etc. Director: A. B. Futo (director of United Chemicals (London) Ltd. and Fubex Ltd.). Reg. office: 9 Arundel Street, London WC2.

PYROMETRIC EQUIPMENT CO. LTD. Cap. £5,000. Manufacturers of and dealers in chemicals and chemical compositions, cement, glues, resins, paints. Reg. office: 49 High Street, Market Harborough, Leics.

J. H. HOLDEN LTD. Cap. £3,000. Wholesalers of chemists' sundries and manufacturers' agents. Directors: S. de Vall, E. de Vall, and K. Bingham. Reg. office: 39 Carver Street, Sheffield 1.

HAMAX LTD. Cap. £10,000. Manufacturers of and dealers in domestic chemical preparations, soaps, cleansers, bleaching fluids and powders. Permanent directors: Frank H. Hamer, Dorothy A. Hamer and Peter R. Hamer. Reg. office: Glasshouse Lane, Mexborough, Yorks.

RELEASE OF RECEIVER

STEWART BROTHERS (LONDON) LTD., chemical, mechanical and electrical engineers, etc. Reigate, Surrey. Mr. J. Wood, 34 Clements Lane, London, EC4, has ceased to act as receiver and manager.

LONDON GAZETTE

Voluntary Winding-up

Notices of companies voluntarily winding-up is purely formal and does not necessarily imply liabilities. It is frequently for purposes of internal reorganisation.

BULSTRODE TECHNICAL SERVICES LTD., research chemists, registered office 16 Great College Street, London SW1. Mr. G. H. Thompson, 833 Salisbury House, London Wall, London EC2, appointed liquidator, 25 September by members.

GOLDEN PRODUCTS (DERBY) LTD., manufacturers of polyoxyethelene monostearate, registered office 119 Alfreton Road, Little Eaton, Derbys. By reason of its liabilities, 25 September. Mr. H. J. Johnson, 120 Green Lane, Derby, appointed liquidator.

E. F. ROSCOE AND CO. LTD., merchants and agents dealing in chemicals and mineral substances, registered office 1 Devonshire Square, London EC2. By special resolution, 19 September. Mr. S. J. Chubb, 4 Tokenhouse Buildings, Kings Arms Yard, London EC2, appointed liquidator.

TRADE NOTES

Epoxy Esters Cheaper

Following the recent price reduction of Epikote, Styrene Co-Polymers Ltd., Sale, Cheshire, announce a reduction in price of the Scopon range of epoxy esters as from 1 October.

Plastics Agency Change

R. H. Cole and Co. Ltd., 2 Caxton Street, London SW1, state that by mutual arrangement with Dunstan Hill Chemicals Ltd., they have taken over the exclusive distribution in the UK of plastics sheet, films and foils manufactured by Rheinische Gummi, Mannheim-Neckarau 17a, Germany.

Wild-Barfield

The telephone number of Wild-Barfield Electric Furnaces Ltd., of Watford, has been changed to Watford 26091.

Telcovin Tube and Sleeving

The Plastics Division of The Telegraph Construction and Maintenance Co. Ltd. (Telcon), Farnborough, Kent, is now producing flexible and rigid p.v.c. tubing and sleeving with the trade name 'Telcovin', to exacting standards. The new product is made in black or white, in colours or in crystal-clear transparent form. Telcovin tubing, having excellent chemical resistance, can be used for the conveyance of mineral oils and petroleum spirit, as well as an almost unlimited range of inorganic chemicals in solution.

Change of Address

Acheson Industries (Europe) Ltd., formerly of 18 Pall Mall, have moved to offices in one of the City's newest buildings—1 Finsbury Square, London EC2 (Monarch 5811).

Technicon Instruments in UK

Technicon International Ltd., New York, have formed a UK subsidiary, Technicon Instruments Co. Ltd., with offices at 26 Warwick Road, London SW5 (Fremantle 5109). The company will manufacture and market in the UK and Commonwealth the AutoAnalyzer for blood chemistry and other Technicon products.

Change of Name

The Marsh Alum Co. Ltd., The Marsh, Widnes, has changed its name to the Viaduct Alum Co. Ltd.

Beneficiated Lectorite

Beneficated magnesium-lithium silicate (Lectorite) is available as Mecaloid from UK agents, Production Chemicals (Rochdale) Ltd., Victoria Buildings, 32 Deansgate, Manchester 3, acting for The Merto Co., San Francisco, US. When fully hydrated Mecaloid will swell from 35-40 fold. Ultimate particle size is stated to be as small as one millimicron thick, 70 millimicron wide and 700 millimicrons long. Ion-exchange capacity (exchangeable ion is sodium) is 100 meg. per 100 grams.

Applications for Mecaloid are: as absorbing agent for proteins, alkaloids, cationically charged organic and inorganic materials; for purifying organic and inorganic waste, and removing colours, and odours from liquids and gases; as flocculating at low concentrations; in formation of strong, stable gels; as suspending agent for pigments, powders and other finely ground materials; as emulsion stabiliser even at autoclaving temperatures; as thick, flexible protective film; and in tabletting.

Uses of Borates in Adhesives

Technical information on the use of borates in adhesives and sizes is contained in Technical Service Bulletin No. 17, produced by Borax Consolidated Ltd. Formulations for the preparation of adhesives indicating the application of borax and derivatives are included. Similarly useful notes are given on the preparation of sizes. The various preparations of borax produced by Borax Consolidated are listed, together with recommendations as to their use.

Farris Valves for UK

Associated Automation, a subsidiary of Elliott-Automation, have signed agreements with both the Farris Engineering Corporation, New Jersey, and the Farris Flexible Valve Corporation. A new company, Farris Engineering Ltd., has been formed to manufacture and sell in the whole of Europe, the British Commonwealth (except Canada) and the Middle East, the Farris range of safety-relief valves and pinch-type 'flex valves'. Flex valves are corrosion resistant and designed for use with slurries and where grit is present.

Spectrophotometer Demonstrated

Mr. K. G. Sölla, of Beckman Instruments, Munich, gave a talk and practical demonstration of the Beckman model IR-5 infra-red spectrophotometer on 24 and 25 September at the London showrooms of Baird and Tatlock (London) Ltd., UK agents for Beckman Instruments Inc. About fifty representatives from various laboratories interested in the instrument attended the sessions.

The model IR-5 is said to be a relatively inexpensive double-beam recording instrument designed for routine qualitative and quantitative analyses in the laboratory. It records linearly in per cent transmittance and wavelength and will scan the entire 2-16 micron range in 16 minutes.

Permatred Flooring

To avoid confusion with the trade name of a roofing material, the heavy duty intensified wood flooring material manufactured by Permali Ltd., Gloucester, is now being registered as 'Permatred' in place of 'Permadec.'

Flexibox Agents

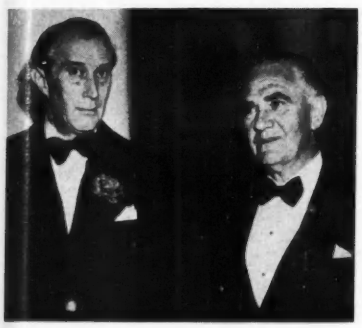
Flexibox Ltd., manufacturers of mechanical seals for rotary shaft equipment, have appointed John Turner and Co., 25 Collingwood Street, Newcastle upon Tyne, 1, as agents for the north-eastern area of Great Britain.

PEOPLE in the news

● MR. R. W. OXTOBY, who has been manager of the Burma Pharmaceutical Industry in Rangoon since its inception, has returned to the head office of Evans Medical Supplies Ltd., Speke, Liverpool 19, on the completion of his contract. He has been appointed director and general manager of Evans Medical (Services) Ltd.

● MR. S. P. CHAMBERS, a deputy chairman of Imperial Chemical Industries Ltd., will give the 10th annual lecture of the Plastics Institute. His subject will be 'International competition in the chemical industry'. Date and venue will be announced later.

● MR. K. W. FRANCOMBE has resigned his position as chief chemical engineer and head of the chemical engineering group of Whessoe Ltd., Darlington. Mr. Francombe, who joined the firm 26 years ago is to devote more time to studying development in the gas and chemical industries and other administrative matters connected with Whessoe. He is succeeded by MR. J. THOMPSON, a former director of APV Engineering Co., Calcutta.



Dr. T. H. M. Kerfoot, president of the Association of British Pharmaceutical Industry, left, with Mr. A. H. Fiske, who, representing the (US) Pharmaceutical Manufacturers' Association was the ABPI's guest at its autumn conference at St. Annes-on-Sea from 1 to 3 October

● DR. C. B. DAVIES, manager of Thornton Research Centre, Shell Research Ltd., since 1952, has been appointed head of the Oil Products Development Department of Shell Petroleum Co. Ltd. in succession to MR. C. HULL. Dr. Davies's successor at Thornton is DR. R. G. LARSON, formerly research director of the Shell Oil Co.'s research laboratory at Martinez, California.

● Three new council members have been elected by the British Welding Research Association. They are DR. R. BEECHING, technical director on the main board of ICI, MR. J. M. WILLEY, director and general manager of Murex Welding Processes Ltd., Waltham Cross, and MR. T. M. HERBERT, director of research, British Railways Division of the British Transport Commission. MR. J. STRONG, this year's president of the Institute of Welding and executive director of British Oxygen Gases Ltd. will represent the institute on the association's council during 1958-9.

● MR. V. F. NOEL-PATON, chairman of T. and H. Smith Ltd., chemical manufacturers, Blandfield Chemical Works, Edinburgh 11, one of the first life barons to be created, has assumed the title of BARON FERRIER OF CULTER IN THE COUNTY OF LANARK.

● DR. E. PARRY-JONES has been appointed deputy chairman of Fison's Pest Control Ltd. He continues as a joint managing director.

● MR. J. P. SIMONS, B.A. (Cantab), has been appointed research fellow in chemistry at Birmingham University. Other appointments made include that of DR. D. HALL, B.Sc., Ph.D. (Dunelm), as ICI fellow in physics.

● Among research fellowships appointed at the annual election of Trinity College, Cambridge University, were: DR. G. A. CHINNER, for research in petrology, and MR. A. D. MCLACHLAN, B.A., for research in theoretical chemistry.

● MR. R. E. HUFFAM, UK co-ordinating director of Unilever Ltd., and chairman of the council of the British Standards Institution, has been elected president of the Institution in succession to SIR H. MANZONI, city engineer of Birmingham. Born in 1894, Mr. Huffam has been in what is now the Unilever group ever since he joined Joseph Crosfield and Sons Ltd., at Warrington, after leaving Gigglewick School in 1913—with the exception of the 1914-18 war years, when he was commissioned in the army and won the MC. He rose from being assistant to the works director of Crosfield's in 1919 to director in 1924 and was appointed chairman of Lever Bros (Port Sunlight) Ltd., in 1930. Eight years later he was transferred to Unilever House, London, as a member of the Home Soap Executive (now known as the Soap Group) and was elected to the board of Unilever Ltd., in 1941. Mr. Huffam is chairman of the British Society of Soapmakers, and has the rare distinction of being an honorary Fellow of the Royal College of Surgeons. His son is a director of the Metal Box Co. Ltd.

R. E. Huffam

● MR. ROBERT HUGH DIBB, M.Sc., M.I.Mech.E., who has been engineering director of ICI Plastics Division since 1946, has succeeded MR. JOHN E. BRAHAM as engineering controller of ICI. Mr. Braham has retired. Mr. Dibb, who is 56, joined the Billingham Division of ICI 31 years ago as a shift manager, and from 1928 to 1939 he rose successively to plant engineer, research engineer and finally to design engineer before he was transferred to the MAP factory at Heysham as deputy construction manager.

R. H. Dibb

● MR. H. E. COOPER, who joined G. A. Harvey and Co. (London) Ltd., London SE7, in 1945 as consulting engineer to the board of management and who was appointed a director in 1946, has become managing director. After serving his apprenticeship, Mr. Cooper became assistant to the chief engineer of Brotherton and Co., Leeds. On taking his B.Sc. he joined the department of the chief engineer, ICI Billingham. At the beginning of the war Mr. Cooper set up a chemical plant in Scotland to manufacture explosives. MR. I. A. MARRIOTT succeeds MR. P. T. BLISS as sales director.

● Celebrating his 40th year with Mobil Oil Co. Ltd., MR. REGINALD W. FOX, coordinator for Lube and allied products in the technical services department, was recently presented with a gold watch and 40-year emblem by MR. BLAKE MIDDLETON, a director.

● MR. B. J. PAGE, secretary and controller, and DR. K. J. LYNES, general works manager, have been appointed directors of Pfizer Ltd., Folkestone.

● VICE-ADMIRAL SIR FRANK MASON has been appointed a member of the Council for Scientific and Industrial Research. A director of Metal Industries and H. W. Kearns and Co., he became chairman of the Mechanical Engineering Research Board last June.

● DR. HAROLD MOORE, C.B.E., has been elected an hon. member of the Iron and Steel Institute, of which he has been a member for 57 years. Until he retired in 1944 he was director of the British Non-Ferrous Metals Research Association.

NEW PATENTS

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Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

ACCEPTANCES

Open to public inspection 12 November

Resinous compositions. Westinghouse Electric International Co. **804 158**

Production of thiourea-aldehyde resins having stable hydrophilic properties and the utilisation thereof. American Cyanamid Co. **804 291**

Producing fertilisers and products thereof. Lummus Co. **804 292**

Process for the production of chloroprene. Distillers Co., Ltd. **804 254**

Production of diphenyl-pyridyl-methanes. Thomae Ges., K. [Addition to 730 243.] **804 159**

Productions of flavoured food compositions. Ruskin, S. L. **804 293**

Apparatus for classifying or settling fluid suspensions. National Research Council. **804 167**

Preparation of curing agents for polyepoxides. Bataafsche Petroleum Maatschappij N.V., De. **804 294**

Lubricating grease compositions. Bataafsche Petroleum Maatschappij N.V., De. **804 256**

Fuel oil compositions containing formaldimine salts. Socony Mobil Oil Co., Inc. **804 305**

Antiparasitic agents. Geigy, AG, J. R. **804 186**

Method for making latex foam rubber. Goodrich Co., B. F. **804 208**

Heat and chemically impervious insulating material and method of preparing same. Connecticut Hard Rubber Co. **804 187**

Preparation of amides of lysergic acid and mixed anhydride useful in such preparation. Lilly & Co., E. **804 188**

Liquid concentrates of sodium pentachlorophenate. Monsanto Chemicals (Australia), Ltd. **804 257**

Method of and apparatus for priming a centrifugal pump for pumping mud, sludge and liquids containing semi-solids. Eisele, J. **804 265**

Fractionator vapour condensing assembly. Universal Oil Products Co. **804 229**

Siloxane-coated articles. Midland Silicones, Ltd. **804 198**

Organo-siloxane compositions. Midland Silicones, Ltd. **804 199**

Iminodibenzyl compounds. Soc. Des Usines Chimiques Rhone-Poulenc. **804 193**

Titanium oxide rectifiers and the manufacture thereof. Yoshida, K. **804 405**

1.3-Di-(1ω-hydroxyalkyl) thioureas. Monsanto Canada, Ltd. **804 299**

1.3-Di-(1ω-hydroxyalkyl) ureas. Monsanto Canada, Ltd. **804 300**

Method of preparing cellulose ethers readily soluble in cold water. Dow Chemical Co. **804 306**

Pharmaceutical preparations for application to the skin or the mucous membranes of the mouth. Opfermann, A. C. J. **804 230**

Production of trifluoroethanol. Pennsylvania Salt Manufacturing Co. **804 231**

Production of sennosides from folia sennae or folliculi sennae. Byk-Gulden Lomberg Chemische Fabrik G.m.b.H. **804 232**

Processes for preparing lubricating compositions. Esso Research & Engineering Co. [Addition to 782 054.] **804 307**

Hydroformylation of olefins. Esso Research & Engineering Co. **804 308**

Method and apparatus for production of liquid hydrogen and oxygen. Garrett Corp. **804 235**

Methods of producing adermin (vitamin B_6). Philips Gloeilampenfabrieken N.V. **804 236**

Methods of producing preparations for combating weeds in flax. Philips Gloeilampenfabrieken N.V. **804 309**

Indole derivative and process for its preparation. Geigy AG, J. R. **804 237**

Ductile chromium and process for preparing same. Du Pont de Nemours & Co., E. I. **804 419**

Chemically expanded rivets and a process for their manufacture. Du Pont de Nemours & Co., E. I. **804 270**

Manufacture of trisubstituted boranes. Olin Mathieson Chemical Corp. **804 341**

Unsaturated diamine products and their preparation. National Distillers & Chemical Corp. [Addition to 802 185.] **804 312**

Fuel composition. National Lead Co. [Addition to 797 391.] **804 278**

Quinuclidines and process for their manufacture. Ciba Ltd. [Divided out of 804 204.] **804 205**

AMENDED SPECIFICATIONS

On Sale 5 November or as soon as possible thereafter

Preparation of organo silicon derivatives from unsaturated compounds. Montclair Research Corp. **684 597**

Organic acid esters of cellulose. Celanese Corp. of America. **779 220**

ACCEPTANCES

Open to public inspection 19 November

Copolymers of perfluoropropene and tetrafluoroethylene and method of making same. Miller, W. T. **804,561**

Production of adhesive bonds between rubber or rubber-like polymers or copolymers and rigid polymers or copolymers. Research Association of British Rubber Manufacturers, Minister of Supply, and Wake, W. C. **804 663**

Propellants. Phillips Petroleum Co. **804 590**

Detectors of oil mists and the like. Gravine Manufacturing Co., Ltd. **804 541**

Preparation of catalysts. British Petroleum Co., Ltd., Porter, F. W. B., and Housam, E. C. **804 593**

Motor gasoline. British Petroleum Co., Ltd., and Fidler, F. A. **804 732**

Methods of working materials made from metal powder. National Research Development Corp. **804 733**

Polymers and catalytic production thereof. Phillips Petroleum Co. [Addition to 790 195.] **804 641**

Production of synthetic linear polyesters. Du Pont de Nemours & Co., E. I. **804 753**

Apparatus for continuously hardening vegetable or animal oils or hydrogenating other non-saturated compounds. Pintsch Bamag AG. **804 604**

Aminopyrine, caffeine or quinine hydrochloride inclusion compounds. Ruben, M. **804 492**

Process for continuously contacting liquids with solids. Socony Mobil Oil Co., Inc. [Addition to 711 487.] **804 632**

Steroids and the preparation thereof. Pfizer & Co., Inc., C. **804 521**

Producing a nitro-acetophenone. Parke, Davis & Co. **804 467**

Treatment of effluents from coal carbonising plants. Gas Council. **804 544**

Proteolytic enzyme. American Cyanamid Co. **804 608**

Preparation of polyalkylene terephthlates and fibres and films made therefrom. Gevaert Photo-Producten NV. **804 495**

Plastic copolymers and articles comprising same. Minnesota Mining & Manufacturing Co. **804 736**

Slip-casting processes. Monsanto Chemicals, Ltd. **804 756**

Manufacture of transparent cellulosic sheets and films. British Cellophane, Ltd. **804 610**

Thermosetting plastisol composition. Sanders (Electronics), Ltd., W. H., and Redfarn, C. A. **804 496**

Mordanting of dyes in photographic layers. Kodak, Ltd. **804 661**

Storage and handling of liquefied gases. British Oxygen Co., Ltd. **804 500**

Fungicidal compounds. Plant Protection, Ltd. **804 444**

Manufacture of solid washing materials containing polyphosphates. Knapsack-Griesheim AG. [Addition to 799 429.] **804 564**

Optical sensitizers. Imperial Chemical Industries, Ltd. **804 670**

Catalytic reforming of petroleum hydrocarbons. British Petroleum Co., Ltd., and Cuddington, K. S. **804 502**

Thermal cracking of liquid hydrocarbons. Chemische Werke Hüls AG. **804 595**

Production of metal oxide catalysts. Badische Anilin- & Soda-Fabrik AG. **804 652**

Glass manufacture. Pemberthy, H. L. **804 505**

Glass compositions and glass-to-metal seals. General Electric Co., Ltd. **804 635**

Whirling apparatus for producing sprays of fluid and for other purposes. Gordon & Co., Ltd., J. **804 446**

Printing or padding of textiles by the pigment printing or padding process. Farbenfabriken Bayer AG. **804 738**

Preparation of an improved fungicide. Vanderbilt Co., Inc., R. T. **804 477**

Preparation of fungicidal supermetallic associates and compositions containing them. Bataafsche Petroleum Maatschappij N.V., De. **804 507**

Dechlorination of organic compounds. Minnesota Mining & Manufacturing Co. **804 508**

Thiol phosphoric acid esters. Farbenfabriken Bayer, AG. **804 761**

Process for the oxidation of organic compounds. Imperial Chemical Industries, Ltd. **804 612**

Antiknock compositions. Ethyl Corp. **804 763**

Alkali metal catalytic compositions. Phillips Petroleum Co. **804 749**

Steroids. Soc. Des Usines Chimiques Rhone-Poulenc. **804 480**

Water-insoluble dyestuffs of the benzene-monoazo-2-hydroxy-3-naphthoic acid anilide series. Farbenfabriken Bayer AG. **804 673**

1-Haloacetoxy-4-substituted-butene. Schwerdle, A., and Schwerdle, M., [trading as Vireland Chemical Co.]. **804 765**

Sensitive materials for colour photography. Kodak, Ltd. **804 662**

Heterocyclic compounds. Farbenfabriken Bayer AG. **804 514**

Metal containing dyestuffs of the benzene-monoazoquinolone series. Farbenfabriken Bayer. AG. **804 766**

Chloroalkane manufacture. Ethyl Corp. **804 515**

Polymerisation of vinyl halides. Monsanto Chemical Co. **804 448**

Incorporation of rubber-like synthetic polymers into bituminous materials. South Eastern Gas Board. **804 556**

Manufacture of γ-(4-chloro-2-methyl-phenoxy)-N-butyric acid and γ-(2:4-dichlorophenoxy)-N-butyric acid. Imperial Chemical Industries, Ltd., Kitchingham, G. W., and Tucker, A. C. **804 565**

Bactericidal compositions. Cadum Palmolive. **804 450**

Glass composition. Pittsburgh Plate Glass Co. **804 451**

Coating metal with polyvinyl chloride. Montecatini Soc. Generale per L'Industria Mineraria e Chimica. **804 516**

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